METALLURGIA

THE BRITISH JOURNAL OF METALS

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METALLURGIA

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INCORPORATING THE METALLURGICAL ENGINEER

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The Duke of Edinburgh's Conference

ONE of the most imaginative experiments of recent years is in progress at Oxford; one which, if it meets with the success its organisers hope for, may have far-reaching effects on the industrial and social life of the peoples of the Commonwealth and Empire. It is just about two years since fifty industrialists and trade unionists were invited, in their individual capacities, to a meeting to advise H.R.H. The Duke of Edinburgh about a suggestion to hold a study conference dealing with the human problems of industrial communities in the Commonwealth and Empire. The matter had been raised with him in his capacity as Patron of the Industrial Welfare Society, on his return from a long visit with H.M. The Queen to many of the countries concerned, and from what he had seen he felt that the idea had much to commend it. His Royal Highness was anxious to find out whether such a conference was thought to be practicable, and to ensure that if it went forward it would do so on lines worthy of such an imaginative conception. The outcome of these and subsequent deliberations has been the organising of H.R.H. The Duke of Edinburgh's Study Conference on the Human Problems of Industrial Communities within the Commonwealth and Empire, to be held at Oxford from 9th to 27th July. His Royal Highness graciously accepted the Presidency of the Conference, and in this capacity agreed to address it at the Opening Ceremony in the Sheldonian Theatre, and to preside at the opening and closing plenary sessions at Rhodes House, and at the two formal dinners at Christ Church.

The Study Conference is an experiment on lines never before attempted in the Commonwealth. It brings together some 280 men and women, mainly within the age group of 25–45, who are engaged in the managerial, technical and operative roles of industry, and who hold, or in the foreseeable future will hold, positions of responsibility, and who have a proven interest in the life of the community. They reflect a wide cross-section of races, industries and occupations, 90 of them drawn from the United Kingdom, 138 from other countries in the Commonwealth, and 52 from colonial territories: in all, 29 countries and territories are represented.

Perhaps the last word of the preceding paragraph is somewhat misleading, because all members have been invited as individuals, and not as delegates, so that they can contribute to the common purpose of the discussions in terms of their own personal experience. This is a Study Conference and its success depends, in the main, on the quality and interest of individual contributions. To this end, the membership has been divided into 20 study groups, which are the working units of the Conference. These groups consist of 14 members, chosen to be representative of a wide range of industrial experience in different countries, and each has a chairman holding a responsible position in industry or the trade unions in different parts of the United Kingdom.

The Conference takes place in three stages. The first, from 9th to 12th July, at Rhodes House, Oxford, takes the form of papers by well-known figures in industry in the United Kingdom, and group discussions. Then each group makes an independent nine-day tour to an industrial centre and to London. These have been arranged so that the group spends as little time as possible simply looking at machinery and processes, and as much as possible in discussion with people, at all levels, inside and outside the factory. Discussions will cover industrial topics and such social problems as housing, education, community life, clubs, health and travel to work. Members will then return to Oxford to hear papers by distinguished persons in industry from overseas, and for further group discussions, the reporting back stage, and the summing-up by Sir Philip Morris, Vice-Chancellor of Bristol University.

The purpose of the Conference is to make a practical study of the human aspects of industrialisation, and in particular those factors which make for satisfaction, efficiency and understanding, both inside industrial organisations and in everyday relations between industry and the community around it. In doing so, the Conference will not deal with matters which come within the normal scope of industrial negotiations. The motives for work, the satisfaction of those engaged in it, the acceptance of discipline and leadership, are profoundly influenced by social and political traditions, by the pattern of family and community life, by the values, fears and aspirations of the individual and of social groups. On the other hand, studies of social development have often emphasized the effects of industrialisation on the traditional life of the community, and these effects have been striking and far-reaching. Industry and social life interact at every point, and it is obvious, therefore, that they can only be studied intelligently as a single whole.

To-day there are, within the Commonwealth and Empire, communities at all stages of industrial development, so that the problems that present themselves are many and varied. We, in the United Kingdom, have long since overcome the problems arising from the industrial revolution: that is not to say, of course, that we have no human problems in industry, as has been forcibly brought home to us recently by the situation in the motor car industry. Again, the more widespread adoption of shift working, which has been suggested as a means of reducing costs sufficiently to enable us to compete in certain world markets, is bound to have its effects on family life and on the social life of the community.

Certain parts of the Commonwealth and Empire are feeling the impact of industrialisation for the first time, and although we and other members of the Commonwealth have been through this phase, and can pass on the benefit of our experience, in each case the peoples

concerned will have their own contribution to make. The industrial revolution of the last century in this country, far-reaching as were its repercussions, was, perhaps, a leisurely sort of transformation when compared with the rate of change made possible by modern technical advances and the annihilation of distance by improved transport and communications. Moreover, in addition to the aspects of industrialisation which affect any community, in some cases further difficulties arise as a result of racial tension.

Obviously then, the Conference gives a unique opportunity to study a common problem against the background of widely differing experience. The significance of the Conference lies in its membership. It is a meeting, not for academic study, but of men and women of many races and nations, who are facing practical responsibilities in industry, and who are prepared to exchange ideas freely and frankly on the tasks and opportunities ahead of them. As the Duke of Edinburgh put it, in the Foreword to the Conference Programme, "Its main value will not lie in the report of the speeches and discussions. Its value will depend upon what the members make of what they see and hear. Its value will lie in the ability of its members to describe their points of view and experience for the benefit of others, and their ability to distinguish what is likely to be useful in their own special cases. Ultimately it is hoped that the members will be able to extend their influence in their own countries and industries, to the end that industrial enterprises are so organised that they form an integral part of a happy and healthy community."

Personal News

Mr. S. R. Howes, who retired as Director and General Manager of Samuel Fox & Co., Ltd., at the end of June, has been appointed a Director of The United Steel Companies, Ltd. He has also agreed to serve for a further three years as consultant for work study and operational research.

Mr. B. Cape has been appointed Technical Services Manager to Kelvin & Hughes (Aviation), Ltd.

Mr. H. D. Boyd, Director of Edgar Allen & Co., Ltd., is at present on a tour of South Africa, Rhodesia, Australia and New Zealand.

Mr. C. F. Lawson has been appointed Foundry Manager of the Distington Engineering Co., Ltd., Workington, and Mr. G. Ellis will act as Personal Assistant to the General Works Manager. Mr. C. Scott has relinquished his position of Assistant Works Manager.

Under the terms of the scheme to commemorate the late Lord Rutherford of Nelson, the Council of the Royal Society has appointed Professor E. N. da C. Andrade, F.R.S., to deliver the Rutherford Memorial Lecture for 1957 in Australia.

The Baird and Tatlock Group of Companies announces that Mr. J. B. Longman has been appointed Technical Sales Representative for South London and South East England in succession to Mr. P. H. D. Andrews who is returning to South Africa.

Mr. G. Smith has assumed the duties of an Assistant Sales Manager to Mr. C. E. Edwards, Commercial Manager of the Appleby-Frodingham Steel Co.

Metropolitan-Vickers Electrical Co., Ltd., announces that Mr. R. F. Marshall has been appointed Manager, Education Department, in succession to the late Mr. K. R. Evans.

Mr. F. N. S. Evans, former Manager, Midland Branch, has been appointed Fuel Oil Manager for the Regent Oil Co., Ltd., and Mr. N. Craven-Jones becomes Manager, Midland Branch.

Mr. T. A. Bird, Sales Manager of the Rolls Division of Armstrong Whitworth (Metal Industries), Ltd., will visit the rolling mills at Pretoria, Vanderbijl Park, and Que Que in Southern Rhodesia. During his tour Mr. Bird will be contacting the Company's agents and giving technical advice and information as part of the normal service to customers.

Vickers, Ltd., announce the following changes in organisation: Mr. A. H. Hird is appointed a Special Director of Vickers, Ltd., and Adviser on Group Commercial Engineering Sales, and also takes over the Chairmanship of A.B.C. Motors, Ltd., Cooke, Troughton and Simms, Ltd., and Ioco, Ltd. He remains on the Board of Vicker-Armstrongs, Ltd., as Engineering Sales Director, and on the Board of Vickers-Armstrongs (Engineers), Ltd.; Mr. E. J. Waddington and Mr. R. P. H. Yapp relinquish their seats on the Boards of Ioco, Ltd., and Cooke, Troughton & Simms, Ltd., and Mr. Waddington relinquishes the Chair of A.B.C. Motors, Ltd., but remains on the Board of that Company; Mr. R. Wonfor is appointed to the Board of Vickers-Armstrongs (Engineers), Ltd., as Sales Director; Mr. J. E. Richardson, O.B.E., is appointed a Director of Vickers-Armstrongs (Engineers), Ltd., retaining his existing office of Deputy-General Manager and Engineering Manager, Barrow Works; and Rear-Admiral Sir Anthony Buzzard, Bart, C.B., D.S.O., O.B.E., is appointed a Special Director of Vickers-Armstrongs, Ltd., and Assistant to the Managing Director.

Obituary

We regret to record the death, on June 21st, 1956, of Mr. J. S. Hollings, C.B.E. Born in 1872, Mr. Hollings was initially trained as a mining engineer, but entered the steel industry in 1893 under J. H. Darby of the Brymbo Steel Co., Ltd., the first man to work the basic open-hearth process in this country. Mr. Hollings was appointed Managing Director in 1914, and when the Company was absorbed by Baldwins, Ltd., he served under another notable pioneer, Sir John Roper Wright, who was associated with Sir William Siemens when the acid open-hearth method of steelmaking was developed. Subsequently, Mr. Hollings served under Sir John's successor, Sir Charles Wright, and later was appointed Managing Director and Vice Chairman of Guest, Keen, Baldwins Iron and Steel Co., Ltd. He retired from this position in 1945, but retained his seat on the Board. During the war his advice was sought by the Ministry of Home Security and his services were recognised by the award of the C.B.E.

Although an acknowledged expert on blast furnace and coke-oven practice, some of his most notable contributions to the industry have been in the field of design and layout of integrated plant. In 1946 he was awarded the Bessemer Gold Medal of the Iron and Steel Institute in recognition of the services he had rendered both to the Institute and to the industry.

The Metallographic Structure of Cast 11–14% Chromium Steels

By Arne Færden

Metallographic studies have been made of cast martensitic stainless steels of two series, one with chromium from 11·3 to 14·1%, and another with nitrogen from 0·011 to 0·056%. The fourteen steels investigated contained from 0·07 to 0·11% carbon, and approximately 0·5% nickel. The steels were first studied in the as-cast and in the tempered conditions, and then the austenitic structure was developed by thermal etching in high vacuum.

STEELS containing 11·5–14% chromium are the simplest types of stainless steel in commercial use. The low carbon type, with about 0·1% carbon, was first produced on a commercial scale in 1920,[1] and is known in Britain as "stainless iron." Martensitic or hardenable stainless steel is the most common name of the same material in the U.S.A.

As chromium has been widely used as an alloying metal in ferrous metallurgy, iron-chromium-carbon alloys have been the subject of many investigations, with results which are not always in complete agreement. Reviews of work on the structure and constitution of iron-chromium-carbon alloys have been written by J. H. G. Monypenny[2]; A. B. Kinzel, W. Craft and R. Franks[3]; C. A. Zapffe[4]; and anonymous authors in Metals Handbook[5]: Fig. 1 is taken from the last-named source.

S. P. Watkins[6] has published a general article on the metallography of stainless steels, including the 11–14% chromium type, and A. E. Nehrenberg,[7] in his article on the transformation of low carbon 12% chromium stainless steels, has reported the variation in amount of *delta*-ferrite with temperature in 12·5% chromium steels containing 0·06%, 0·10% and 0·12% carbon. The latest work in the 11–14% chromium region is by R. L. Rickett and his collaborators,[8] who publish an equilibrium diagram (Fig. 2) which shows some modifications compared with those of Tofaute, Sponheuer and Bennek,[9] and Tofaute, Küttner and Büttinghaus.[10]

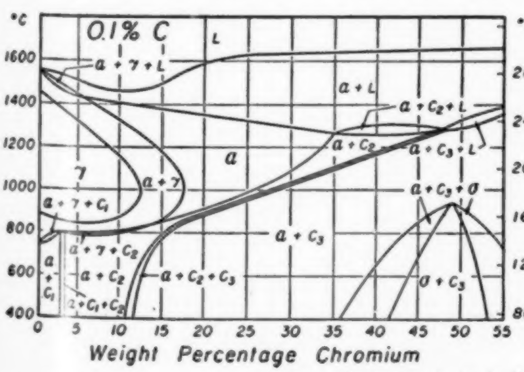
A complete understanding of the constitution of commercially produced stainless steel calls for the modification of the iron-chromium and iron-chromium-carbon equilibrium diagrams for pure alloys (Figs. 1 and 2) to take account of the effect of other elements. It is well known that carbon, nitrogen, nickel and manganese expand the austenitic range, in contrast to chromium, silicon, molybdenum and aluminium, which contract it.

A survey of the literature shows that relatively little

TABLE I.—COMPOSITION OF STEELS

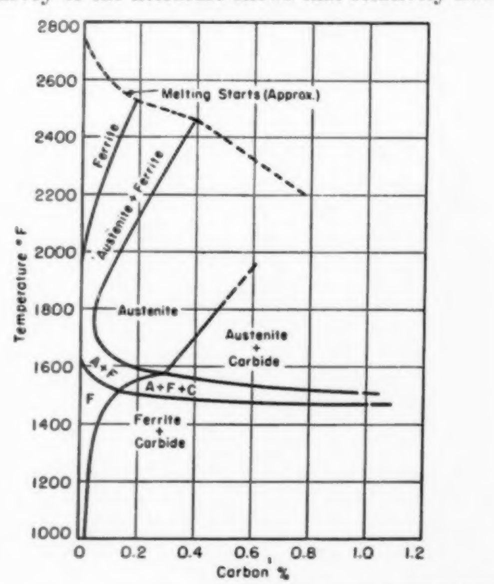
Steel	Composition (%)								
	Cr	Ni	Mn	Si	C	N	S	P	Ti
A	11·3	0·54	0·61	0·35	0·10	0·014	0·002	0·014	*
B	12·2	0·40	0·65	0·34	0·11	0·015	0·005	0·017	*
C	12·6	0·67	0·63	0·60	0·12	0·011	0·003	0·019	0·005
D	13·1	0·63	0·64	0·30	0·11	0·033	0·003	0·015	*
E	13·3	0·57	0·63	0·36	0·14	0·020	0·003	0·025	*
F	13·8	0·50	0·70	0·26	0·11	0·027	0·003	0·017	*
G	14·1	0·68	0·64	0·48	0·10	0·032	0·007	0·019	0·01
H	12·2	0·48	0·56	0·40	0·07	0·011	0·002	0·016	*
I	12·8	0·48	0·57	0·54	0·09	0·019	0·003	0·020	*
J	12·4	0·58	0·68	0·44	0·10	0·024	0·002	0·019	*
K	12·2	0·57	0·62	0·39	0·11	0·028	0·003	0·013	*
L	12·5	0·58	0·65	0·44	0·10	0·034	0·003	0·015	0·01
M	13·1	0·97	0·47	0·43	0·07	0·056	0·004	0·016	0·01
N	12·3	0·59	0·63	0·47	0·11	0·028	0·003	0·012	*

* Less than 0·004%.



Courtesy of the American Society for Metals

Fig. 1.—0·1% carbon section of the iron-chromium-carbon equilibrium diagram.[5]



Courtesy of the American Society for Metals

Fig. 2.—12% chromium section of the iron-chromium-carbon equilibrium diagram.[8]

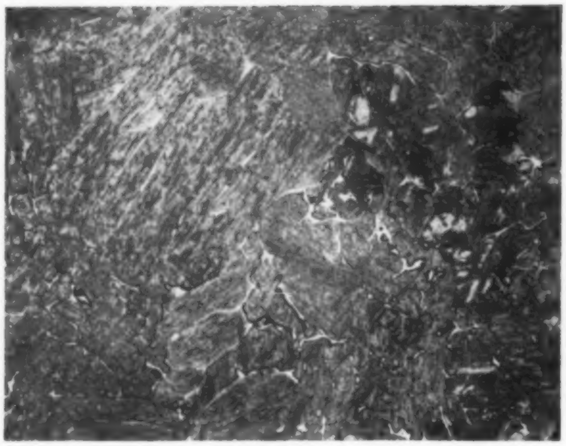


Fig. 3.—Steel N, as cast. Etched Vilella's reagent. × 50.

Fig. 4.—Steel N, as cast. Etched Vilella's reagent. × 300.

systematic work has been reported on the metallographic structure of 11–14% chromium steels with low carbon, and that nothing has been published on these steels in the "as cast" condition.

Experimental Details

Materials

All the steels used were made on a commercial scale in an electric arc furnace. Those listed in Table I were all cast in sand moulds to give bars of about 40 kg. weight. The metallurgical conditions under which different charges were made were very much the same, with the exception of steel *M*, where poorer deoxidation conditions may be assumed.

From Table I, it will be seen that the steels in this investigation cover a considerable range of composition, particularly with respect to chromium and nitrogen. The percentage chromium increases from 11·3 in steel *A* to 14·1 in steel *G*, whilst nitrogen increases from 0·011% in steel *H* to 0·056% in steel *M*. To maintain the concentration of the other elements as constant as possible, the various steels in Table I were selected from a total of about fifty.

Heat Treatment

Apart from *N*, all steels were first heat treated at 970° C. and then at 750° C. In both cases the specimens were heated in an electric furnace with a heating rate of 50° C/hour, held at temperature for six hours and cooled in air.

Preparation and Etching of Specimens.

The standard polishing procedure used involved grinding by hand on emery paper, down to 4/0 grade, after which the specimen was further ground on a rotating wheel carrying broad cloth soaked in paraffin wax and impregnated with 600 mesh Carborundum powder. For final polishing, magnesium oxide was used, and the specimen was then deeply etched in Vilella's reagent :—

Hydrochloric Acid ..	10 ml.
Picric Acid	2 g.
Ethyl Alcohol (96%) ..	200 ml.

* Unless otherwise stated, the term "heat treated" in captions of the photomicrographs refers to the following treatment : Heating at 970° C. for 6 hours, air cooling, tempering at 750° C. for 6 hours and again air cooling.

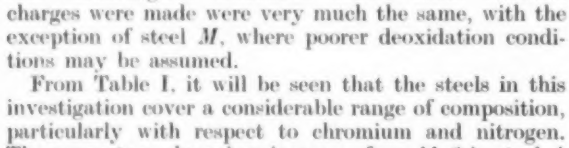


Fig. 5.—Steel N, as cast. Thermally etched at 970° C., for 2 hours and "air cooled." × 150.

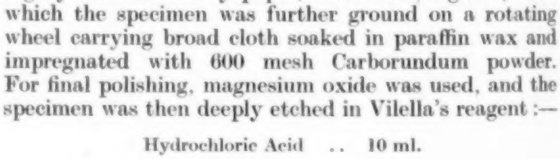


Fig. 6.—Steel F, heat treated.* Etched Vilella's reagent. × 100.

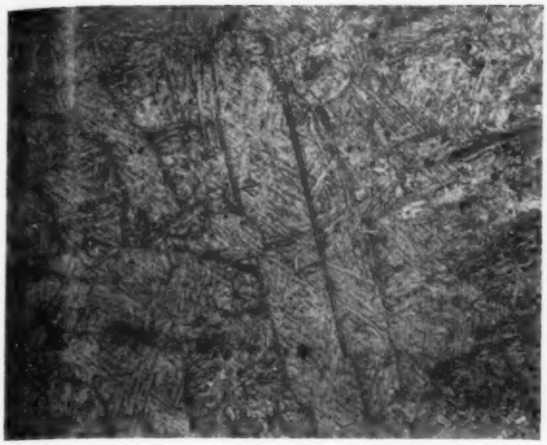


Fig. 7.—Steel G, heat treated. Etched Vilella's reagent. ×100.

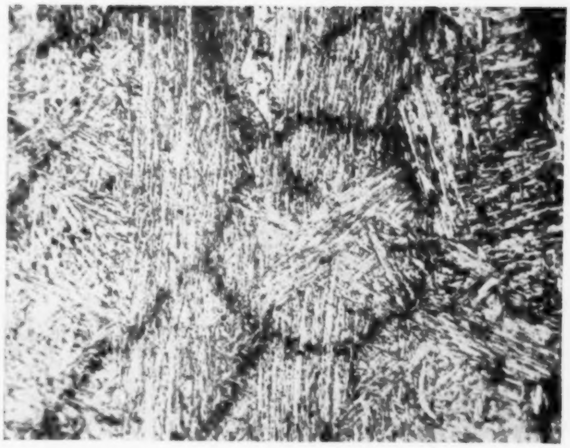


Fig. 8.—Steel J, heat treated. Etched Vilella's reagent. Note the "dotted lines." ×200.

and repolished three times.* After a final repolishing, the specimen was etched in Vilella's regent for 60–120 seconds.

When the specimens had been studied in the as-cast or tempered condition, they were all repolished and thermally etched at a definite temperature for a definite time.

As-Cast Structure

When steels such as those listed in Table I pass from the liquid to the solid state, the last liquid to freeze has a more ferritic composition than the average composition of the molten steel before freezing commenced. This is clearly shown in Fig. 3, a specimen from steel *N* (12·3% chromium, 0·11% carbon). The course as-cast pearlitic grains are outlined by ferrite (white) and, as is to be expected, there is no sign in Fig. 3 or Fig. 4 (at a higher magnification) of the previous austenitic grain size.

The specimen of steel *N* was then thermally etched† at 970° C. for two hours and "cooled in air." The austenite grains developed in this way are shown in Fig. 5, from which it will be noted that they are outlined by ferrite. Furthermore, the austenite grain size is larger than that developed during the heat treatment.

As *N* was the only steel not heat treated when received, it was the only one which could be investigated in the as-cast condition.

Tempered Structure

Specimens from the remaining steels (*A–M*) had all been given the heat treatment described (970° C. for six hours, air cooled, tempered at 750° C. for six hours, and air cooled). In addition to the general structure of tempered martensite—as seen in Fig. 6—formation of ferrite, "dotted lines," and traces of former austenitic structure were observed.

As published equilibrium diagrams for pure alloys cannot be used to predict the structure of commercial 11–14% chromium steels, R. I. Rickett[8] published a tentative "structural" diagram, based on steels from his investigations. Using the same factors as Rickett, good agreement between carbon content, "equivalent"

* Some time after the completion of the experimental work, a paper by A. E. Ehrenberg[11] was published, in which he recommended the following solution for etching 12% chromium steels: 0·25–0·5% hydrochloric acid by volume in 50 ml. of 5% picral. This etchant shows good contrast between the tempered martensite and *delta*-ferrite in hardened and tempered martensitic steels.

† This method will be described briefly in the section on "Austenitic Structure."

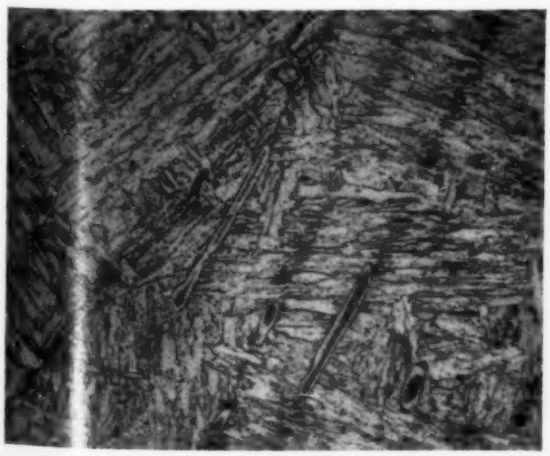


Fig. 9.—Steel J, heat treated. Etched Vilella's reagent. See Fig. 8. ×300.

Fig. 10.—Steel B, heat treated. Etched Vilella's reagent. ×300.

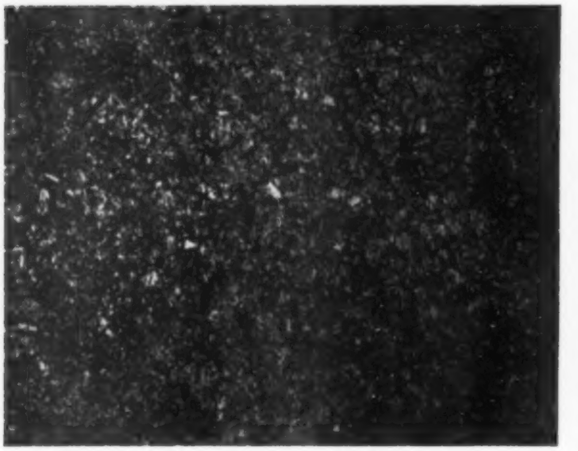


Fig. 11.—Steel A, heat treated. Thermally etched at 970° C. for 10 hours and "air cooled." × 50.

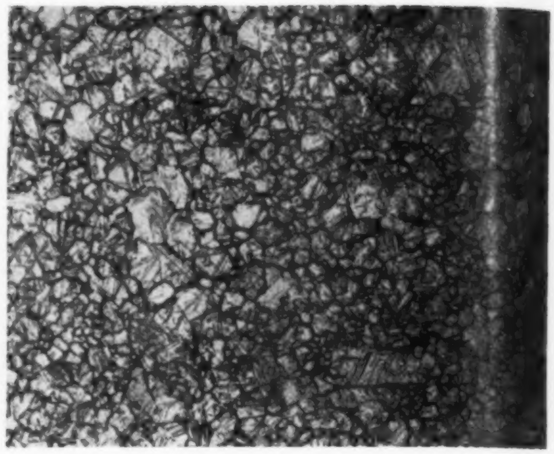


Fig. 12.—Steel A, heat treated. Thermally etched at 1,020° C. for 10 hours and "air cooled." × 50.

chromium content, and ferrite present, was found in the steels used in this investigation.

At a magnification of × 100, there was no evidence of ferrite in steels *A* and *B* after tempering, and specimens from steels *E*, *F*, *K* and *L* contain very little ferrite, as shown in Fig. 6. The amount of ferrite has increased in steels *C* and *J*, and still more in *D*, *H* and *I*. When the amount is even higher, it becomes characteristic for the tempered structure (and for the high temperature structure revealed by thermal etching, as will be shown later) as may be seen from Fig. 7, a specimen from steel *G*.

On almost every specimen a pattern of dotted lines was found, similar to that seen in Fig. 8 (steel *J*), from which it is possible to see that the dotted lines also consist of long ferrite islands. This is seen more clearly in Fig. 9, taken from the same specimen. It seems probable that the dotted lines are traces of the ferrite seen between the pearlite grains in the as-cast structure (Fig. 3).

As for the as-cast structure, no sign was found of the austenitic structure formed during the first cooling. When the steels were heated to 970° C., austenite was again formed, and traces of this austenitic structure were seen in all specimens investigated in the tempered condition. The former austenite is seen in Fig. 9, but more distinctly in Fig. 10, a specimen from steel *B*.

Austenitic Structure

All methods of revealing the size of the primary austenitic grains depend upon the fact that grain boundary material, in general, exhibits greater chemical and physical activity than do the grains themselves.

The Mc-Quaid-Ehn, or carburisation, method was the first to be employed to reveal austenitic grain size in steel. This work stimulated a considerable number of researches, and today several methods are known. Of these, the thermal etching, fracture, and martensitic etch methods are the only ones which do not introduce carbon and/or oxygen into the steel. Additions of such elements radically change the composition, and an austenitic structure revealed in a steel thus altered may be entirely different from that which is characteristic of the original metal at the same temperature revealed by one of the three methods mentioned above.

Reviews of the various methods of revealing the austenitic grain size have been published by N. F. Ward and J. E. Dorn,[12] B. R. Nijhawan and A. B. Chatterjee,[13] and, for cast steels, by M. J. Hawkes.[14] Later work on this subject has been reported by E. J. Eckel and S. J. Paprocki,[15] who used a method based on intergranular diffusion of bronze into steel at elevated temperatures, and by M. J. Olney,[16] who used thermal etching.

The last-named method has been used mainly in the present investigation, but the fracture and martensitic etch methods were also tried. The apparatus used for high vacuum etching was that described in detail by M. J. Olney.[17] The etching was carried out on previously polished specimens, which were placed in a silica tube sealed at one end and continuously evacuated by means of a mercury diffusion pump. The pressure—unless otherwise stated—did not rise above 5×10^{-4} mm. of mercury, as measured on a McLeod gauge. In all experiments, the silica tube containing the specimens was heated in a tube furnace fitted with a controller. Heating was normally for two hours at 970° C.

To obtain information about some of the variables which are believed to have an effect on the austenitic structure of 11–14% chromium steels developed by thermal etching in high vacuum, the following factors were investigated :—

(1) Austenitising temperature and time.

(2) Rate of cooling.

This investigation also contributed further information on :—

(3) Influence of steel composition on austenitic structure.

(4) Fracture at low temperature.

(5) Vilella's reagent for revealing austenitic grain size.

(6) Formation of twins.

Before detailing the results of this work, reference should be made to the difficulty of expressing numerically the range of the austenitic grain size. This arises from the great difference in the size of grains in cast steel, as was stressed in an earlier paper by M. F. Hawkes.[14]

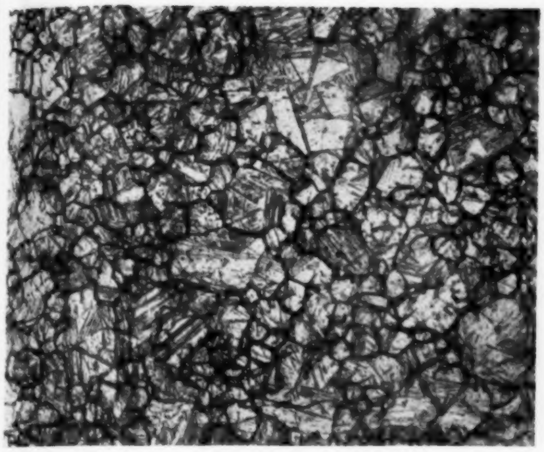


Fig. 13.—Steel A, heat treated. Thermally etched at 1,045° C. for 10 hours and "air cooled." × 50.

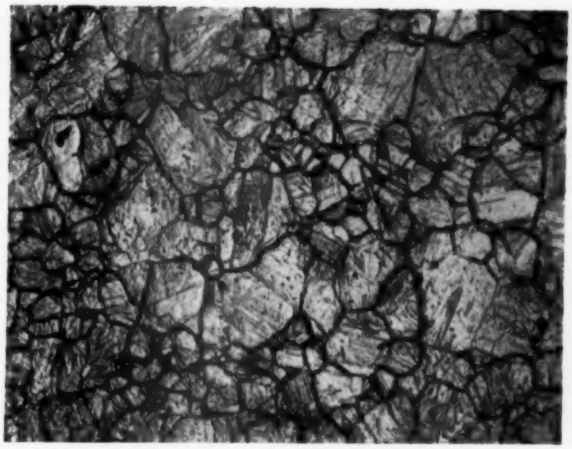


Fig. 14.—Steel A, heat treated. Thermally etched at 1,070° C. for 10 hours and "air cooled." × 50.

Austenitising Temperature and Time

In investigating the influence of temperature in the *gamma* region, the following temperatures were used: 870, 920, 970, 1,020, 1,045 and 1,070° C. Specimens from steel *A* did not show any sign of grain growth when heated for ten hours at 870, 920 or 970° C., but, when the temperature was increased to 1,020° C. marked grain growth occurred, and still more at 1,045 and 1,070° C. Grain growth with increasing temperature is shown in Figs. 11–14.

R. L. Rickett and his collaborators[8] recorded grain growth in a rolled 12% chromium steel, containing 0·11% carbon, when heated in the temperature range 870–970° C. When they heated the steel to 1,095° C., however, they found less grain growth than has been found in the present investigation.

Specimens with high chromium content (14·1%), from steel *G*, were heated in vacuum to 920° C. (Fig. 15) and 970° C. (Fig. 16). As might be expected, the ferrite (white) is more evident in the former: the austenite grains are further outlined by ferrite, and are smaller in size than in the specimen heated to 970° C.

As far as the influence of time at temperature is concerned, steels heated for ten hours showed little difference in grain size from steels heated for two hours at the same temperature.

Rate of Cooling

After heating in high vacuum for a definite temperature and time, the specimens were usually "air cooled" under vacuum with the furnace removed. In addition to this procedure—referred to in this paper as "air cooled"—quenching in mercury and slow cooling with the furnace in position were also tried.

The appearance of the high temperature vacuum etched surface is very much the same whether quenched in mercury or "air cooled." In both cases, the outlining of *gamma* grains is to some extent disturbed by formation of martensite needles—see, for instance, Figs. 16 and 20—but the needles never cross the grains or the twin boundaries of the austenite.

If the rate of cooling is so slow that no, or very little, martensite transformation takes place (see Fig. 17[8]), the

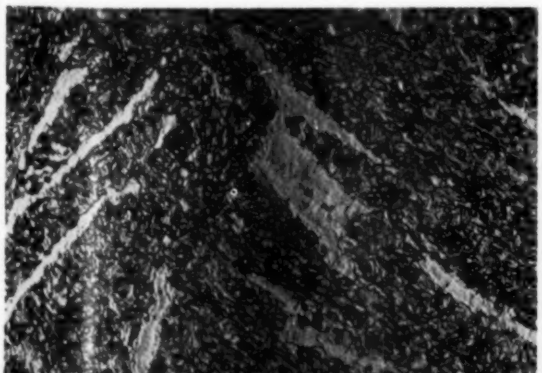
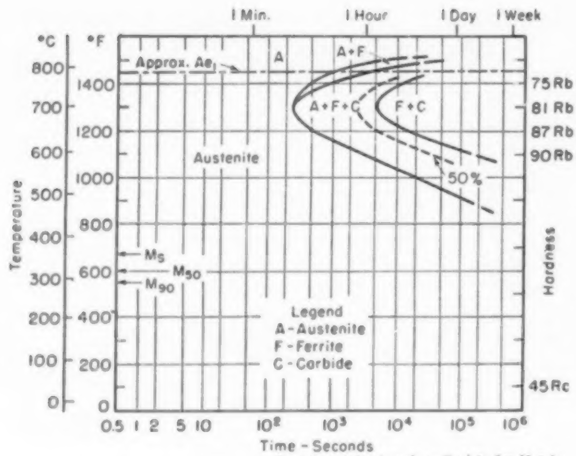


Fig. 15.—Steel G, heat treated. Thermally etched at 920° C. for 2 hours and "air cooled." × 150.

Fig. 16.—Steel G, heat treated. Thermally etched at 970° C. for 2 hours and "air cooled." × 150.



Courtesy of the American Society for Metals

Fig. 17.—Isothermal transformation diagram for a representative 12% chromium steel.[8]

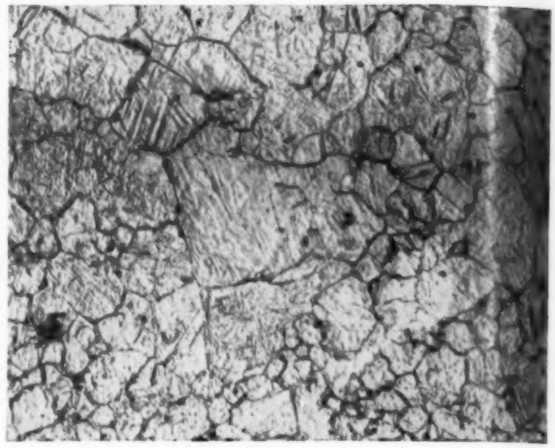


Fig. 18.—Steel E, heat treated. Thermally etched at 970° C. for 2 hours and furnace cooled. ×150.

outlining of the *gamma* grains is much clearer—as shown in Fig. 18 from steel *E*. The specimen was thermally etched at 970° C. for two hours and slowly cooled inside the furnace. This is in disagreement with the work of M. J. Day and J. B. Austin,[17] who found that interpretation of the thermal etch patterns was facilitated if the specimens were quenched in mercury.

Unfortunately, as the slow cooling was very time-consuming, it was not possible to use it in the limited time available and, in all other cases in this paper, specimens were "air cooled."

Composition of the Steels

To investigate the influence of nitrogen, and of chromium and nitrogen, on the austenitic grain size, specimens from steels *A–M* (Table I) were all heated in vacuum at 970° C. for two hours and then cooled in air.

The effect of increasing the nitrogen content from 0·011% in steel *H* to 0·034% in steel *L* seems to be small, but with higher nitrogen contents the austenitic grain size is somewhat smaller. Steel *M*, with 0·056% nitrogen (added as high nitrogen ferro-chromium), 13·1% chromium, and 0·07% carbon, would be expected to have this smaller grain size. It is, however, believed that this steel was less efficiently deoxidised than the rest, and poorer deoxidation and, perhaps, a more ferritic composition may explain why it shows a coarser grain size than expected. In general, the effect of nitrogen in reducing the grain size in 12–14% chromium steel is in agreement with the work of R. Franks,[19] but the effect is less than expected.

The influence of chromium was investigated in steels with chromium contents ranging from 11·3% in steel *A* to 14·1% in steel *G*. From earlier determinations of oxygen in 12–14% chromium steels, it is known that the oxygen content tends to decrease with increase in chromium.

Nitrogen tends to some extent to reduce grain size, and from Table I it will be seen that steels with a higher chromium content also have a higher nitrogen content. If the compositions of the steels were such that 100% austenite formed at elevated temperatures, it would,

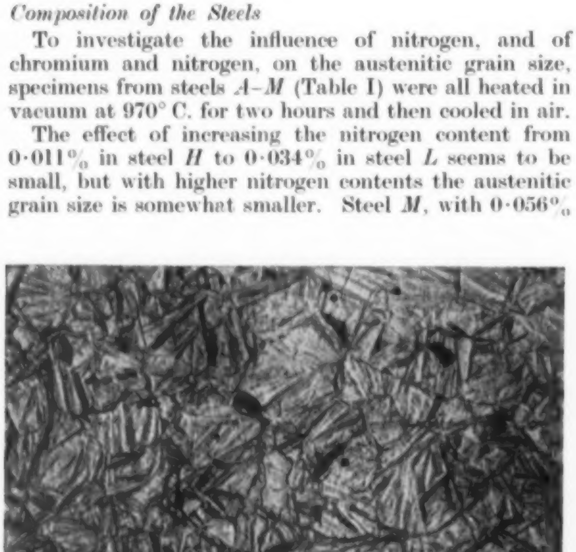


Fig. 19.—Steel A, heat treated. Thermally etched at 970° C. for 2 hours and "air cooled." ×150.

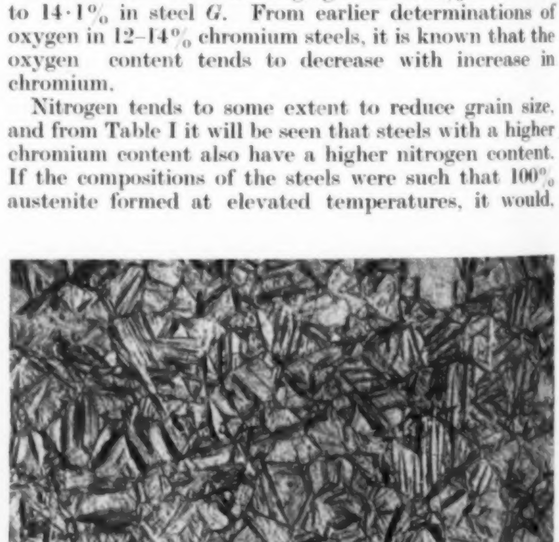


Fig. 20.—Steel D, heat treated. Thermally etched at 970° C. for 2 hours and "air cooled." ×150.

Fig. 21.—Steel D, heat treated. Thermally etched at 970° C. for 2 hours and "air cooled." ×500.

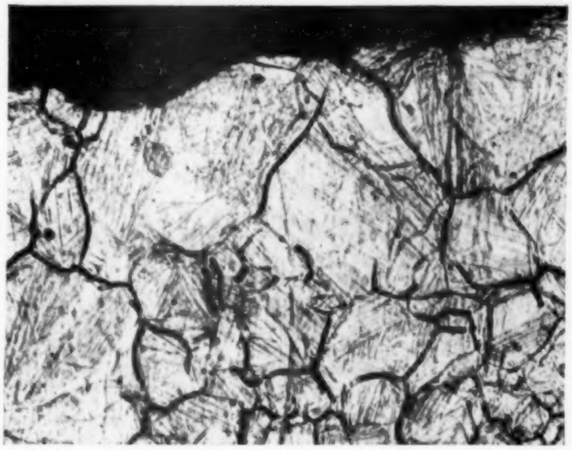


Fig. 22.—Steel A, heat treated. Thermally etched at 1,045° C. for 10 hours and "air cooled." Fractured in liquid oxygen. ×150.

therefore, be reasonable to believe that the austenitic grain size would be somewhat smaller with higher chromium content. With still higher chromium content, ferrite will be stable at elevated temperature (970° C.): ferrite then becomes characteristic for the structure.

Fig. 19 shows the austenitic grain size of steel *A*, with 11·3% chromium: in Fig. 20, a specimen from steel *D* (13·1% chromium), the grain size is somewhat smaller. With still higher chromium content, ferrite becomes characteristic for the structure, as can be seen from Fig. 16 (steel *G*, 14·1% chromium).

Concerning the indication of the former austenitic structure on thermally-etched specimens, only a few were put on record. One of these is shown in Fig. 21, where grains from the former austenitic structure may be seen (just a little to the right of the centre of the photomicrograph).

Fracture at Low Temperature

Notched specimens from steel *A*, first thermally etched at 920, 970 and 1,045° C. for ten hours, and then "air cooled," were fractured in liquid oxygen. All specimens gave a typical ductile fracture, and Fig. 22 shows the structure of that heated to 1,045° C.

This was not at all unexpected, as M. F. Hawkes[14] has found that the fracture method of revealing austenitic grain size fails to work at all in about one-third of the cast steels (carbon and low alloy) which he studied. It happens quite often that specimens exhibit a ductile fracture, even though they are 100% martensite. This ductility is not unusual in low carbon martensite, and it extends to higher carbon contents as the alloy content of steels is increased.

As fracturing in liquid oxygen results in ductile failure, the method cannot be used for revealing austenitic grains in martensitic stainless steels.

Vilella's Method

Vilella's method for revealing austenitic grains was also investigated, but it was not possible to get clear

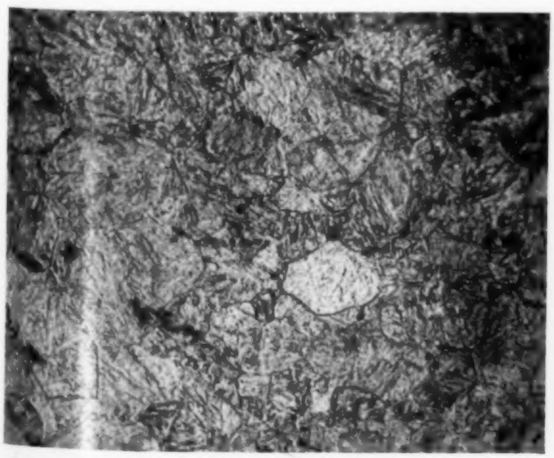


Fig. 23.—Steel A, heat treated and then heated at 970° C. for 2 hours and quenched in mercury. Etched in Vilella's reagent. ×300.

Fig. 24.—Steel D, heat treated. Thermally etched at 970° C. for 2 hours and furnace cooled. Note the twins. ×800.

outlining of the grains. This is shown in Fig. 23, which refers to a specimen of steel *A* which, after polishing, was heated in vacuum for 2 hours and then quenched in mercury. The specimen was etched and repolished several times. It should be pointed out that time did not allow detailed studies to be made with this method, and it is, therefore, possible that, if the right conditions were worked out, the outlining might be satisfactory.

Formation of Twins

It is known that austenite, in common with other face-centred-cubic metals, twins on (111) planes, and observations of austenitic twins have been described previously by M. J. Olney.[17] In the present work, twins were frequently observed on thermally-etched specimens, as may readily be seen from a specimen of steel *F* heated in vacuum at 970° C. for two hours and slowly cooled. Twins are also seen in Figs. 5, 12, 13, 14, 16 and 20. Details of an austenitic twin in a specimen of steel *D*, which was thermally-etched at 970° C. for two hours and slowly cooled, are shown in Fig. 24.

Acknowledgments

This work was carried out in the Department of Metallurgy, in the University of Cambridge. The author wishes to express his thanks to Professor G. Wesley Austin for his great interest, encouragement and advice, and to Mr. G. C. Smith for many stimulating discussions. Thanks are also due to Mr. A. B. Winterbottom for information and helpful discussions.

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Introduction to Work Study

WORK study is the application of detailed analysis to work. The first formulation of its principles dates back as far as forty years, and isolated applications have been successfully pioneered during the whole of the intervening period. Only in the past five years, however, has the realization spread that the basic discipline of work study and the attitude of mind that it generates enable it to be applied to a wide variety of activities in industry, commerce and other fields. It is not confined to highly repetitive operations, but can profitably be applied to jobbing, maintenance and many other kinds of work in which there is little or no repetition. Recent applications have led to improved and more productive methods in such divergent quarters as agriculture and the retail store. Work study is universal; it can be as useful in the household kitchen as it is on the factory floor.

Growing Interest

A measure of the growing interest in the subject during the past few years has been the steady demand for the general introduction to work study first published by the British Institute of Management in 1951. Under the title "Outline of Work Study—Part I: Introduction" this B.I.M. booklet is now re-issued* in a completely revised form, incorporating recent experience, and brought into line with current wider thinking. Part II of this series, a volume devoted to method study, was published in October, 1955. A third volume, entitled "Work Measurement" will be published shortly. In the writing of this series, grateful acknowledgment is made to the directors of I.C.I., Ltd., who have generously made available material used in connection with the company's work study courses.

The object of the present introductory volume is to give the non-specialist general manager all the background information that he needs as to the principles of work study, the purposes for which it can be applied and the results that may be expected. Chapters devoted to method study and work measurement, the two complementary branches of work study, foreshadow the more detailed study of these subjects in the later volumes. A final chapter on work study and management clearly outlines the manager's responsibility in this field. "Work study," it says, "is no more a substitute for good management than it is for technical knowledge and experience in the different specialist functions. Provided this is continuously borne in mind, the various techniques comprise one of the most powerful tools at management's disposal in the day-to-day business of developing and directing an organisation." At the same time "in itself work study can achieve nothing. For success it depends entirely upon what use is made of it by responsible management."

Management's Role

In a foreword to the booklet, a warning is issued to management on three points connected with work study. These are: firstly, that the conviction of its value and the drive to apply it must come from the top; secondly, that management must make clear to all concerned what it is trying to do and why; and thirdly, that, although work measurement is an essential part of work study, it is vitally important that methods should be analysed and improved as far as possible before any attempt is made finally to measure and set standards for the various jobs concerned.

* "Outline of Work Study—Part I: Introduction" is obtainable from the British Institute of Management, 8, Hill Street, London, W.1. Price 3s. 6d.

BRITISH INSULATED CALLENDER'S CABLES, LTD., announce that as from 1st July the sole telephone number for their Cambridge depot is Cambridge 55451-2.

Marker Movement in Scale Layers

By K. Sachs, M.Sc., Ph.D., A.I.M.

G.K.N. Group Research Laboratory

Nickel and copper markers, in the form of electro-deposits and wires of various thicknesses, were placed on the surface of mild steel specimens which were subsequently oxidised at 900° C. The position of the markers in the scale was determined and it was found that during oxidation thick wires move outward, very thin markers move inward, and intermediate sizes occupy intermediate positions. Copper markers move further inward than nickel ones of corresponding size. The various mechanisms which may account for this behaviour are discussed.

PFEIL[1] demonstrated the counter-current diffusion of iron and oxygen in the oxidation of iron and steel by the gradual growth of the scale round inert particles placed on the orginal surface of the metal; he also showed that surface features of the original specimen were accurately reproduced in the scale, and that the position of these features was unaffected by the progress of oxidation. If iron oxidised by the inward diffusion of oxygen and its reaction at the metal surface, inert markers would remain on the surface of the scale; if the mechanism of oxidation depended exclusively on the diffusion of iron through the oxide lattice and chemical reaction at the surface of the scale, the bodily movement of the oxide as it followed the receding metal core would retain inert markers at the surface of the metal core. The position of the inert markers at the original metal-oxide interface implies counter-current diffusion of both iron and oxygen. Since iron has considerably smaller atoms than oxygen, its diffusion coefficient was expected to be higher than that of oxygen. Nevertheless the amount of iron diffusing outward through the scale is kept down to a value corresponding to the amount of oxygen moving inward by the porosity formed in the oxide; this reduces the effective cross-section of diffusion path available to iron, while oxygen can move through the pores by dissociation of the oxide and gaseous diffusion, as well as through the oxide lattice by ionic diffusion.

More recently, Smigelskas and Kirkendall[2] have discovered that inert markers may move through a metal lattice owing to difference in the mobility of two interdiffusing metals. The distances involved were smaller than could be detected by Pfeil's method of determining the position of inert markers in the scale, i.e., chemical analysis of mechanically separated layers. This has led to a re-examination of marker movement in scale layers. The most important investigation was that of Davies, Simnad and Birchenall,[3] whose very elegant experimental technique involved the application of very small quantities of radioactive silver to the surface of iron prior to oxidation. They found the markers at the metal-oxide interface, and concluded that wüstite grows almost entirely by diffusion of iron ions, while oxide ions are added at the outer face of the phase and remain essentially fixed. On the other hand, Brasunas and Grant[3, 4] have confirmed Pfeil's observation that inert markers at the original iron/atmosphere interface do not move. Brief references to the measurement of marker movement also occur in a paper by Dunnington, Beck and Fontana,[5] who found that the markers had moved away from the metal [illegible] of the thickness of the wüstite layer.

These contradictions in experimental observations involve distances of marker movement far in excess of those observed in metals, where agreement between different investigators is far closer (*cf.* references 2 and 8). For this reason, they are not readily explained by inaccuracies of measurement, and must be attributed to mechanisms operative in the scale which obscure or interfere with the Kirkendall effect. The present work shows that volume changes and the porous nature of the scale should lead to deviations from the anticipated marker positions which it is easier to predict than demonstrate experimentally, and that the size and composition of the markers has a profound effect on their movement through scale, unlike their behaviour in metallic diffusion.[8]

Marker Movement in Metal and in Oxide Lattices

The theoretical study of marker movement in a metal lattice due to the interdiffusion of two metals differing in diffusion coefficient starts with the assumption that there is no change in volume. This does not apply to the growth of oxide layers, and consideration of marker movement in scale might well start with a calculation of the number of iron and oxygen atoms present in that part of the inner scale layer bounded by the original interface.

It is convenient to consider a portion of the scale having flat parallel faces at both the metal/oxide and wüstite/magnetite interfaces, a base area of 1 sq. cm., a thickness of ξ cm., the part within the original interface being ξ' cm. thick. Before oxidation, the number of iron atoms within volume ξ' cu. cm., which is, of course, the total number in the final oxide, is given by the formula $\frac{4\xi'}{a^3}$, where a is the parameter of γ-iron, i.e., $3{\cdot}63 \times 10^{-5}$ cm. at 1,100° C.[6] After oxidation, the number of atoms in the oxide lattice can be expressed as $\frac{\xi'(1-p)}{b^3}$, where p is the porosity as a fraction of the volume, and b is the distance between neighbouring iron and oxygen atoms on the cube planes of the wüstite lattice; b varies across the wüstite layer with the iron concentration, from $2{\cdot}140$ to $2{\cdot}155$, and the mean value used in the calculation is $2{\cdot}147 \times 10^{-8}$ cm.[7] It is assumed as a first approximation that the porosity is sufficiently uniformly distributed through the relevant portion of the scale to be expressed as a fraction of the total volume.

Assuming the average composition of the non-stoichiometric wüstite phase to correspond to the formula $Fe\,O_{1{\cdot}13}$, the number of iron atoms remaining inside the original interface will be $\frac{\xi'(1-p)}{2{\cdot}13\,b^3}$, so that the

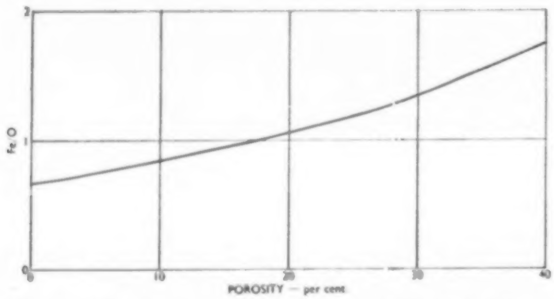

Fig. 1.—Effect of porosity on ratio of ions crossing original interface.

number of iron atoms which have diffused into the outer scale layers is $\frac{4\xi'}{a^3} - \frac{\xi'(1-p)}{2\cdot 13\, b^3}$. The number of oxygen atoms within the original interface is $\frac{1\cdot 13\,\xi'(1-p)}{2\cdot 13\, b^3}$. The oxygen in the pores can be neglected.

The ratio of the number of iron atoms which have moved outward across the interface, to the number of oxygen atoms which have moved inward can be calculated from these formulae, by inserting arbitrary values for the porosity. The ratio is plotted against the porosity in Fig. 1. In scales with little porosity the number of oxygen atoms within the original interface is greater than the number of iron atoms which have left this volume. In spongy oxide layers, on the other hand, more iron has moved outward than oxygen inward across the original interface.

Inert markers placed at the original metal/atmosphere interface should finally occupy a plane across which equal numbers of iron and oxygen atoms have diffused in opposite directions. If Pfeil's suggestion is applied, that the porosity keeps down the transport of iron ions to that of oxygen ions, and if there is no change in volume, this plane will remain at the original interface, but in oxides the markers will move a distance $\delta\xi'$ from the position of the original interface. The scale in the volume bounded by the markers will contain $\frac{(\xi' + \delta\xi')(1-p)}{2\cdot 13\, b^3}$ iron atoms and $1\cdot 13 \frac{(\xi' + \delta\xi')(1-p)}{2\cdot 13\, b^3}$ oxygen atoms. The number of iron atoms which have diffused outward past the markers is $\frac{4\xi'}{a^3} - \frac{(\xi' + \delta\xi')(1-p)}{2\cdot 13\, b^3}$, and this has been assumed to be equal to the number of oxygen atoms which have passed the markers in the direction of the metal core. Under these conditions

$$\frac{\delta\xi'}{\xi'} = \frac{4b^3}{(1-p)\,a^3} - 1$$

The distance between the original interface and the new position of the inert markers can be expressed as a fraction of the thickness of the inner scale layer (within the original interface) for different values of the porosity. The results are plotted in Fig. 2. In the absence of porosity the markers would move inward; in a scale with about 18% porosity the markers would remain at the initial interface; at higher porosities the markers would move outward. The distances moved in scales with reasonable porosity are sufficiently small to be within the limits of error of Pfeil's observations. The validity of these considerations obviously requires experimental confirmation.

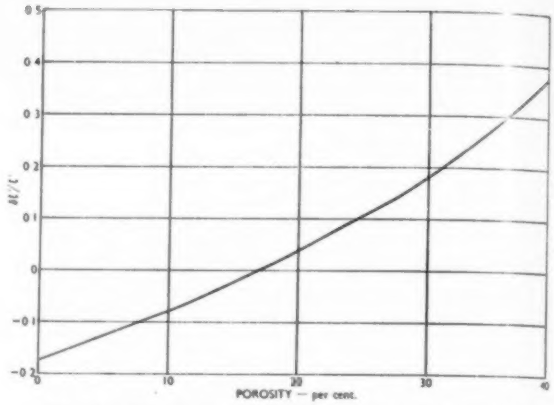

Fig. 2.—Effect of porosity on marker movement.

Size and Nature of the Markers

The significance of the Kirkendall effect in metal lattices was confirmed by experiments which showed that similar results could be obtained with different types of marker.[8] It was felt that experiments with various markers in oxide lattices might help to explain the contradictions between the results obtained by different investigators. The work was undertaken in the belief that qualitative indications of the main factors controlling marker movement could be gained by a limited number of simple and rather crude experiments. Experimental results must, therefore, be interpreted with extreme caution, and should not be regarded as fully established until they can be confirmed by more rigidly controlled experiments.

Experimental Procedure

Apart from a few experiments with copper wire which had been platinum plated, most of the work was done on mild steel. Short lengths of $\frac{1}{8}$ in. diameter mild steel welding rod were cut off, provided with markers, and placed on porcelain crucibles in an air muffle at about 900° C. for various periods. The weight gain due to oxidation was recorded and, in general, conformed to the parabolic equation. Copper and nickel were used as markers, in the form of electro-plate and wire. The welding rod carried a copper flash and this served as the marker in one series; it was removed and replaced by an equivalent nickel flash in another. Somewhat thicker electro-plate of the two metals (0·003–0·005 cm. thick) was employed in another two series. Thin wire of copper or nickel 0·020 cm. diameter, was twisted in a very open helix round the specimens, so that a cross-section of the specimen also revealed a cross-section of the wire, while the markers were free to move laterally during oxidation (*cf.* reference 5).

Bigger specimens were required for experiments using rather thick wires as markers. Standard 0 B.A. nuts were employed; these are made from a free-machining mild steel. The nuts were placed on a few parallel short lengths of copper wire (0·120 cm. diameter) or nickel wire (0·200 cm. diameter); more pieces of the wire were placed on top and held in position by a light frame. In other experiments similar specimens and markers were

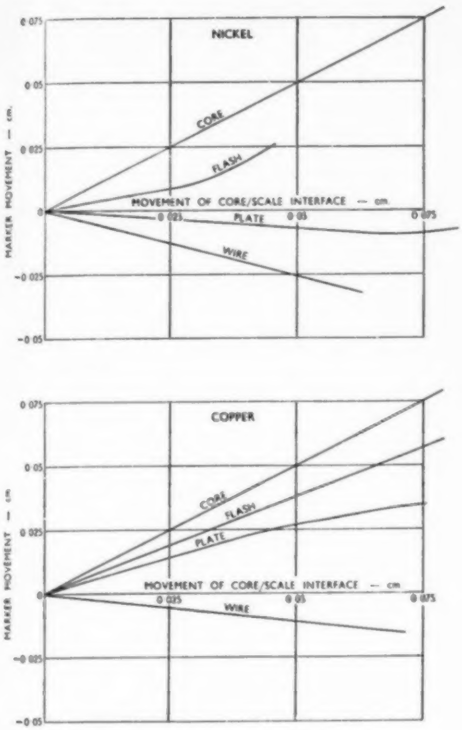

Fig. 3.—Movement of different types of marker.

held in position by a screw through the centre of the specimen which gripped the nut and wire markers between two washers. In this set-up the mechanical force on the markers was fairly high initially, but decreased rapidly due to relaxation of the screw and oxidation of the washers. Nevertheless, outward movement of the markers was restrained, access of air to the marked surfaces of the specimen was restricted by the washers, and the experimental conditions correspond to restrained movement of the markers at slow rates of oxidation.

The oxidised specimens were immersed in a solution of Diakon in chloroform and then dried. The solution filled all but the smallest pores in the scale and the plastic left behind after drying increased the mechanical strength of the scale sufficiently for the specimen to be mounted in Diakon at the usual pressure. The mount was then sectioned and prepared for micro-examination. The dimensions of the metal core and between the markers were measured on a Vickers Projection microscope.

Experimental Results

The movement of the markers in the scale formed on short lengths cut from mild steel welding rod is plotted against the contraction of the core in Fig. 3. Three oxidation experiments were carried out with each type of marker, the time at temperature varying between 8 and 156 hours. The results for each series are reasonably consistent, and from the appearance of the curves it can be concluded that the experimental error is not likely to exceed ± 0·01 cm.

Oxidation of mild steel wire carrying a very thin

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Fig. 4.—Copper particles near iron core of mild steel wire oxidised for 98 hours at 900° C. ×450.

nickel flash produced a scale containing some metal particles close to the core/scale interface and a very small number of isolated particles further out in the scale. These latter particles had a faintly yellowish tinge and probably consisted of nickel. Their location is shown in the upper graph of Fig. 3; there has been a distinct inward movement of nickel from the original metal surface.

The copper flash is broken up into smaller and more numerous particles than the nickel, and they are closer to the core than the corresponding nickel particles, as indicated in the lower graph of Fig. 3. These copper particles are intermingled with the iron particles close to the core/scale interface; under the microscope they can easily be distinguished by their colour, and in photomicrographs (Fig. 4) by their darker shade.

A nickel electro-deposit 0·005 cm. in thickness slows down oxidation in the early stages. After 8 hours at 900° C. the diameter of the core had only shrunk by 0·004 cm. against 0·010 cm. with a nickel flash, 0·015 cm. with copper plate of similar thickness, and 0·021 cm. with copper flash; this trend persists over longer periods, but gradually becomes less pronounced as the deposits lose cohesion and are separated from the metal/oxide interface. As a result of the very slight oxidation, no movement of the nickel plate marker was observed in the specimen heated for 8 hours at 900° C. The nickel plate already contained sufficient oxide to provide a path for the oxidation of the base metal. At a few points the nickel plate had separated from the underlying metal to form small blisters, and oxidation of the iron had progressed further at these points than elsewhere. The blister may have formed by separation of the nickel plate due to surface stresses during heating up. As oxidation progresses, the electro-plate is undermined and prised away from the surface of the core. Two representative blisters are shown in Fig. 5, and the biggest one found on this specimen in Fig. 6. Since the blisters are not filled with oxide, stress must have played a large part in their formation.

In similar specimens oxidised for longer periods, the nickel plate assumes a very irregular outline. This is illustrated in Fig. 7 for a specimen oxidised 156 hours at 900° C. The experimental points for nickel-plated specimens in the upper graph of Fig. 3 represent the mean

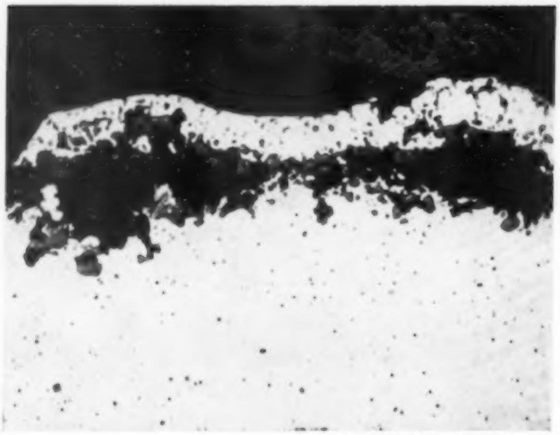

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Fig. 5.—Nickel plate on mild steel wire oxidised for 8 hours at 900° C. × 30.

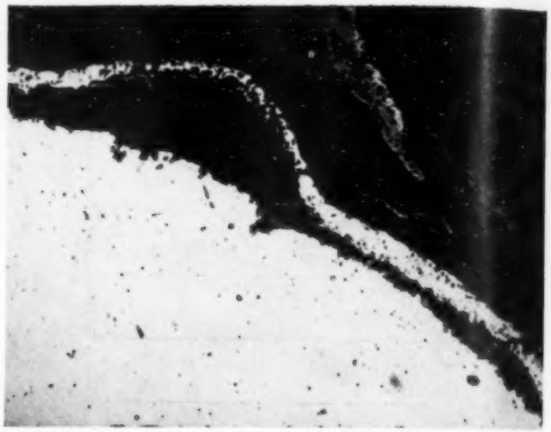

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Fig. 6.—Nickel plate on mild steel wire oxidised for 8 hours at 900° C. × 150.

of 15 measurements across different diameters. However, the local deviations on either side of the original interface greatly exceed the mean outward movement indicated by this graph. Spalling of the plate due to surface stresses is not likely to be a predominant factor; the difference in magnitude between the stress-produced blisters in Figs. 5 and 6 and the gross serrations in Fig. 7 can only be accounted for by a mechanism operating over a fairly long period; stresses in the nickel deposit would not persist for long at 900° C.

Serrations of intermediate magnitude were observed in copper specimens carrying a very thin electro-deposit of platinum (Fig. 8). The mean of eight measurements indicated an inward movement of 0·005 cm. after 63 hours and an outward movement of 0·003 cm. after 156 hours at about 900° C. The variations between different readings exceeded these values, as was the case with the nickel-plated steel.

Mild steel wires with a copper deposit about 0·005 cm. thick were oxidised under similar conditions. Copper particles were found in a wide band extending from the core into the scale layer. The width of the band was measured and is plotted on the lower graph of Fig. 3. The copper particles were coarsest at the outer edge of the band and became gradually finer toward the iron core. Fig. 3 shows an appreciable inward movement of the markers.

Thin wire markers are difficult to hold to the surface of the unoxidised specimen, so that a small gap between the marker and the original interface may be an unavoidable feature of this type of specimen. The corresponding curves in Fig. 3 indicate, however, that other factors, such as the progress of oxidation and the nature of the wire, have sufficiently consistent effects not to be masked by the random variation due to this gap. The copper wires were broken up into particles, some of which were near the iron core. The sites of the original wires were occupied by a vermicular aggregate of copper particles, located at distances plotted in Fig. 3.

In the experiments with small mild steel nuts and thick wires, the " markers " were found on the outside surfaces of the scale; this is shown in Figs. 9 and 10 for specimens oxidised 60 hours at 900° C.; the copper wires in Fig. 10 are completely oxidised but the copper oxide layer is at the outer surface of the iron oxide scale. Thick wires held in place with screws and washers did not move from their original positions. The copper wires were broken up into particles scattered in the scale.

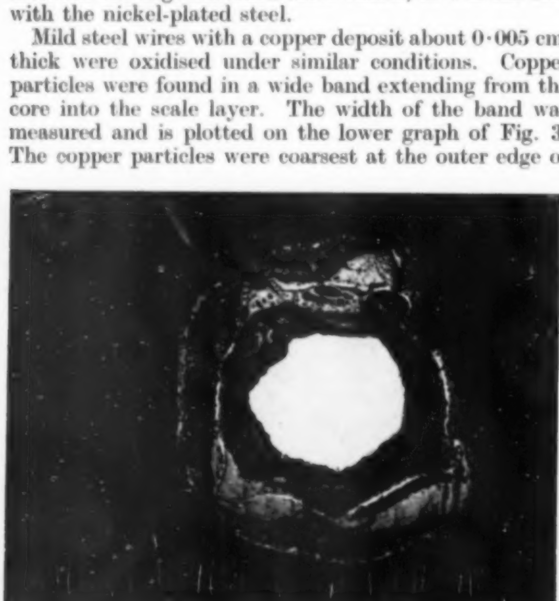

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Fig. 7.—Nickel plate on mild steel wire oxidised for 156 hours at 900° C. × 13.

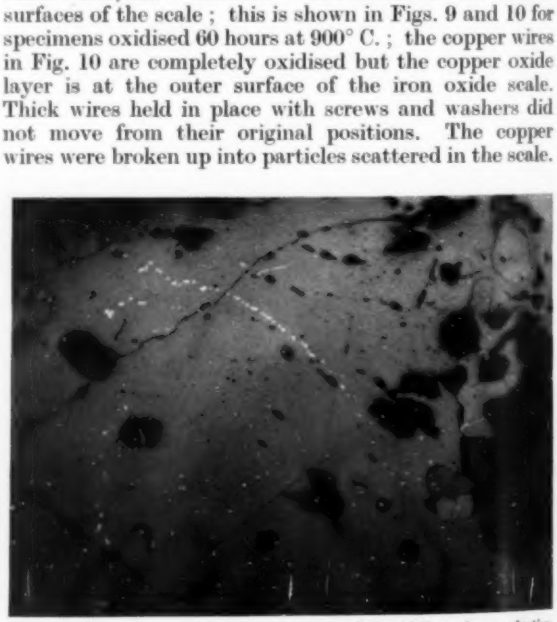

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Fig. 8.—Platinum plate on copper wire oxidised for 63 hours at 900° C. × 200.

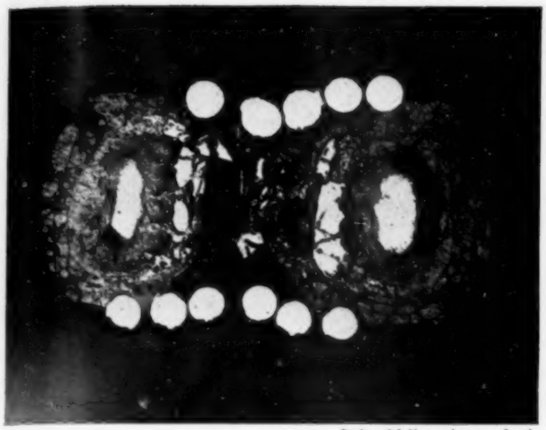

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Fig. 9.—Thick nickel wire on mild steel nut oxidised for 60 hours at 900° C. × 5.

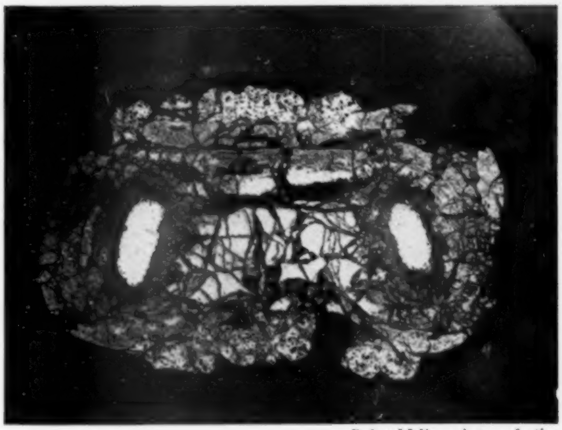

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Fig. 10.—Thick copper wire on mild steel nut oxidised for 60 hours at 900° C. × 5.

Copper particles between the screw shank and the washer are illustrated in Fig. 11, and Fig. 12 shows copper particles in the gap between the screw head and the washer; it is clear that copper atoms have moved over considerable distances through the scale.

In order to establish that the observed marker movements recorded in Fig. 3 are not fortuitous, an experiment was carried out with the object of demonstrating that after oxidation copper markers are closer to the core than nickel markers. A short length of mild steel welding rod was stripped of its copper flash, stopped off on half its circumference and copper plated on the other half; the stopping-off paint was removed, the copper plated portion was stopped off and the other half-circumference of the wire was nickel plated. Short specimens of this wire were oxidised for various periods at 900° C. One copper/nickel joint on a specimen oxidised for 48 hours is illustrated in Fig. 13. The copper particles close to the core are shown at a higher magnification in Fig. 14, and the nickel particles, which are evidently a little further away from the core, in Fig. 15.

Discussion

Marker movement in oxide scales evidently depends on the nature and size of the markers. Markers consisting of very small particles of copper or nickel (flash deposit after oxidation) move inward toward the core, very thick wires of these metals move with the outer surface of the growing scale, and intermediate sizes move to intermediate positions in the scale. At each size except the largest, the copper markers are nearer the core than the corresponding nickel markers.

It is obvious that, in some cases at least, the Kirkendall effect is overshadowed by other mechanisms which give rise to marker movement over much greater distances. Two such mechanisms will be suggested, to account for the outward movement of very large markers on the one hand, and the inward movement of very small ones on the other. Large markers interfere with diffusion by obstructing the path of migrating atoms. In studies on the Kirkendall effect in metal lattices,[8] it was found that foil markers curl at the edges, i.e., that diffusion takes place round the edges of the foil. In the case of oxidation, large markers act as barriers, not only to the counter-current diffusion of iron and oxygen, but also to the formation of the oxide lattice in which diffusion takes place. Those parts of the surface which are not covered by the barrier oxidise in the normal way, but beneath the barrier oxidation is possible only as a result of lateral diffusion, and is very slow; the formation of

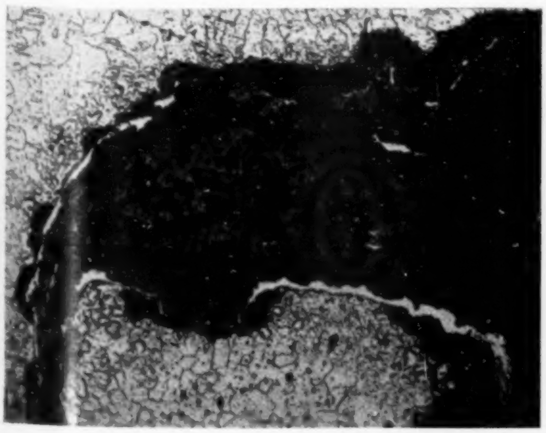

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Fig. 11.—Copper particles in gap between screw and washer. × 75.

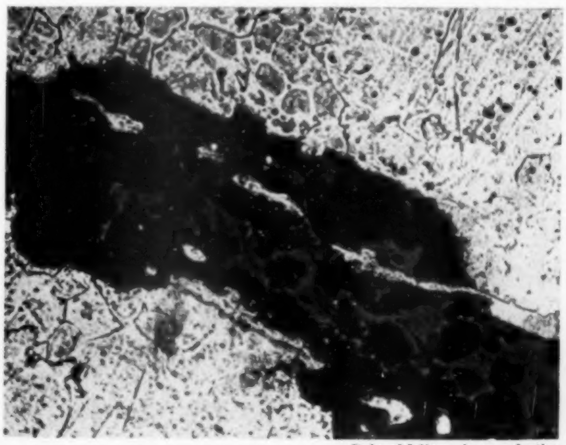

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Fig. 12.—Copper particles in gap between screw and washer. × 400.

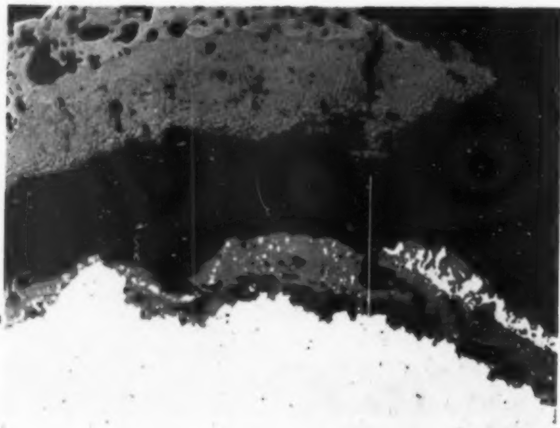

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Fig. 13.—Joint of copper and nickel plate on mild steel wire oxidised for 48 hours at 900° C. ×100.

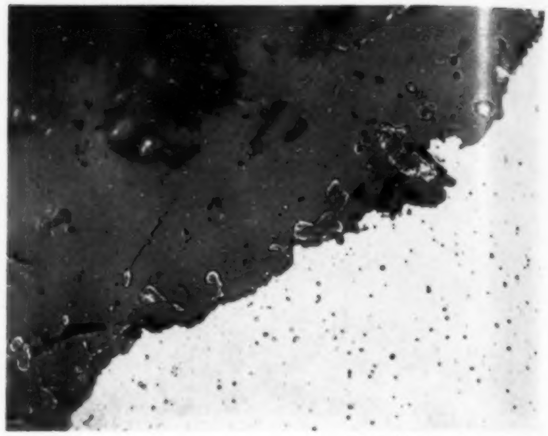

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Fig. 14.—Copper plate on mild steel wire oxidised for 48 hours at 900° C. ×500.

oxide under the barrier lifts the barrier off the specimen. Thus, markers which are large enough to form barriers to oxidation will be forced outward from the original interface. This effect will operate until the barrier is pierced by oxidation or completely enveloped in oxide growing all round it.

The persistence of the barrier effect will depend on the size of the marker and its tendency to oxidation. The thick wire markers, for instance, were never surrounded by scale and, therefore, acted as effective barriers throughout the period of oxidation, so that all the oxide was found beneath the barrier, i.e., the markers remained on the outer surface of the scale. The thinner wires presumably were also kept on the surface of the growing scale, but the oxide growing freely on the adjacent surface of the steel soon reached a thickness where it closed in over the wires and formed an oxide/atmosphere interface outside them. The outward movement of the marker would probably continue at a slower rate until the length of diffusive path round the marker became negligible compared with the distance from the surface of the scale. This would suggest a decrease with time in the velocity of outward travel. In this connection it must be emphasised that the lines drawn on the graphs in Fig. 3 serve only to connect corresponding experimental points, and are not intended to suggest a mathematical relationship. At 900° C. the copper wires oxidise more rapidly than the nickel markers, so that the barrier effect operates for a shorter time and the outward movement of the markers is less pronounced.

The possible operation of the barrier effect in nickel plated specimens has already been mentioned in the description of the scaled samples. It would not affect the entire electro-deposit, only small zones which have not been pierced by oxide particles. These small flakes might be expected to finish at an angle to the core/scale

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Fig. 15.—Nickel plate on mild steel wire oxidised for 48 hours at 900° C. Near joint with copper plate—copper particles have precipitated on the outer surfaces of nickel particles. ×500.

interface, and the barrier effect might considerably enlarge blisters originally formed by stress spalling. The irregularity in outline was more pronounced with the nickel plate than with the much thinner platinum deposit on copper specimens, where the barrier effect would presumbly cease much earlier. The barrier effect can have had no significance in cases where the markers were found to move inward.

Migration of markers towards the core was most pronounced when copper was used. Figs. 11 and 12 show the general tendency of copper to diffuse toward iron and " plate out " at or near the surface, under conditions where copper is evidently stable in the metallic state. It is suggested that the thermodynamic stability of metallic copper and nickel in wüstite scale does not mean that the metal remains completely inert, but that the reduction of the copper or nickel oxide to metal

$$Cu_2O + Fe \longrightarrow 2Cu + FeO$$

$$NiO + Fe \longrightarrow Ni + FeO$$

is more rapid than the reverse reaction. It is assumed that both reactions take place, resulting not in the formation of copper or nickel oxide but in the movement of copper or nickel atoms through the wüstite lattice toward higher iron concentrations and their precipitations as metal particles. Although wüstite does not dissolve copper or nickel, atoms of these metals may be regarded as defects in the wüstite lattice which can migrate through it and precipitate at suitable nuclei.

If it is assumed that this kinetic equilibrium effect operates, it can account for the inward movement of small copper and nickel markers. Larger markers will not move bodily, but will diminish in size, and some small particles will be distributed throughout a band bounded by the markers and the core/scale interface. It is suggested that copper exhibits greater mobility than nickel by this mechanism, and, for this reason, in the present experiments the copper flash and plate moved inward, while in the case of nickel only the thin flash migrated ; sufficient of the nickel plate remained *in situ*, without being involved in alternate oxidation and reduction, to serve as inert markers which were influenced by other mechanisms already discussed.

Conclusion

It would appear that the specimens in which the influence of the barrier and the kinetic equilibrium mechanisms was least, so that marker movement depended primarily on the different rates of counter-current diffusion of metal and oxygen, were those with nickel plate and copper wire markers. These remained close to the original interface, as did the inert markers used by Pfeil[1] and by Brasunas and Grant.[3, 4] Markers which are not " inert " from the point of view of the Kirkendall effect may be pushed outward mechanically (barrier effect), or may diffuse through the oxide lattice and precipitate as small particles near the metal core (kinetic equilibrium effect). The observations of Davies, Simnad and Birchenall,[3] who found minute silver markers close to the metal core, suggest that their silver markers went into forced solution in the oxide, that the silver diffused over long distances through the oxide lattice and finally plated out on the iron core.

In general, the movement of markers is influenced by several mechanisms, the direction of movement depending on the size of the markers, the extent of movement on their chemical constitution. No conclusions concerning the relative importance of the diffusion of iron or oxygen ions through the scale can be drawn unless other mechanisms are eliminated, e.g., the markers are truly inert. The present results indicate that Pfeil's original suggestion of counter-current diffusion of both iron and oxygen is substantially confirmed.

Acknowledgment

The author is indebted to Dr. T. Emmerson, Director of Research, for permission to publish this paper and to Mr. F. D. Bridge, Manager of the Metallurgy Division, for fruitful discussions and advice.

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Restoring Titanium Ductility

THE costliness of titanium often makes the reclamation of the fabricated metal important, both for reasons of economy and time. Titanium alloys are used in the manufacture of forged compressor wheels for the United States J 73 aircraft jet engine : such wheels are now machined from two titanium alloys—Ti-140A and RC-130B.

Before acceptance, the wheels are required to pass a minimum tensile specification. The Aircraft Gas Turbine Division of the General Electric Company found that the rejection rate for some of the Ti-140A wheels was running above 80%, because of insufficient ductility. This rejection rate made reclamation highly desirable. Battelle was asked to study methods of treating the wheels so that their ductility would be increased above the specified minimum. Institute metallurgists were also asked to attempt to develop parallel treatments for both types of forgings.

D. N. Williams and R. I. Jaffee of Battelle report that heat treatments were developed which appreciably improved the ductility of both the Ti-140A and RC-130B compressor wheels. In the treatment giving the best results for Ti-140A, the material was annealed about 100° F. (55° C.) below the *beta transus* (about 1,600° F. (870° C.)). It was then slowly cooled (2° F./min. (1·1° C./min)) to the stabilising temperature of 1,200° F. (650° C.), where it was held for a short time. This resulted in a large increase in ductility with but a slight loss in strength. A parallel treatment was developed for RC-130B. In cases where a high hydrogen content was present in Ti-140A, heat treatment in vacuum resulted in a remarkable improvement in thermal stability.

Correction

RECENT HEAT TREATMENT FURNACE INSTALLATIONS. Fig. 38 on page 271 of the June issue showed equipment made by Delapena and Son, Ltd., for hardening working and bearing surfaces of camshafts, using high-frequency heating. We regret that this was shown with the axis of the camshaft in the horizontal position, whereas in fact the equipment operates with the camshaft vertical.

The Embrittlement of Tungsten by Ammonia

By G. L. Davis, B.Sc., Ph.D., A.R.I.C., F.I.M.
Materials Research Laboratory, Mullard, Ltd.

Heating tungsten wires to high temperatures in atmospheres containing small concentrations of ammonia results in pronounced embrittlement. In the present paper, the author outlines the results of an investigation of the conditions governing this phenomenon, from which he draws a number of interesting conclusions.

TUNGSTEN metal oxidises rapidly above 600° C., so it is processed and used at higher temperatures *in vacuo* or in protective atmospheres of inert or reducing gases. Pure nitrogen is satisfactory for its inertness, and hydrogen for its reducing properties, but it is generally advantageous to mix the two gases, for the mixtures are less explosive and have a lower thermal conductivity than hydrogen, while any residual oxygen is more easily removed than from nitrogen alone. When the component gases are pure, mixtures of them are completely satisfactory for atmospheres in which to heat tungsten wires, but it is well known in the industry that traces of impurities such as oxygen, water vapour or carbon-containing gases can cause severe embrittlement of the wires.

During an investigation into brittle wires, it was discovered that small concentrations of ammonia gas could have a disastrous effect on the toughness of tungsten wires heated in a mixed gas (90% nitrogen : 10% hydrogen) atmosphere, and in view of the prevalence of nitrogen-hydrogen mixtures derived from dissociated ammonia, it was considered that the subject warranted some study.

The embrittling might be due to ammonia as such, or to nascent nitrogen and/or hydrogen formed by the dissociation of ammonia at the hot surface of the metal —although no reference to such an effect on tungsten from these three gases has been found in the literature. In the absence of more direct information, some degree of chemical interaction of the metal with these gases might be significant, but the findings of different workers are not altogether in agreement. Nevertheless, there is some evidence that tungsten reacts with nitrogen[1-7] and ammonia[8-10] at very slow rates to form products (imide, nitride), which tend to decompose above *c.* 950° C.[9], so that the observed sensitivity to these gases above 1,000° C. was rather unexpected.

Experimental

For the tests, a typical sample of commercial tungsten wire, of diameter 250μ, was cut into suitable lengths. Heating was effected by passage through the specimens of alternating current taken from a variable transformer, and was carried out in a vessel containing the chosen atmosphere at atmospheric pressure. This atmosphere was a mixture of nitrogen with 10% hydrogen (which had been freed from oxygen and water vapour by passage through the usual trains, with phosphorus pentoxide as the final desiccant) to which ammonia had been added. The temperature of the wire was determined by an optical pyrometer, and the readings were corrected for the spectral emissivity. These wires were tested as-received from the drawing machines (without pre-treatment), and were still coated with the film of graphite lubricant which is used in the drawing process ; this film is rapidly removed in the reducing atmosphere used, and has no embrittling effect when heating is carried out in ammonia-free gas.

Ammonia was introduced into the atmosphere in controlled amounts by passing the gas stream over pure ammonium chloride, held at a controlled temperature, and then immediately through a bed of heated soda-lime. The concentration of ammonia was determined for a measured volume of gas by absorption in a given volume of N/10 sulphuric acid and subsequent back-titration with N/10 caustic soda. Ammonia concentrations from 0 to 10% by volume were readily obtained, and the apparatus was calibrated for ammonia concentration against furnace temperature for subsequent operation. The calibration curve so obtained was found to be reproducible at all stages of the experimental work.

" Brittleness " is inherently a somewhat subjective term, and a quantitative measure is rarely easily made. After ammonia treatment, the brittleness of tungsten wires was patently evident in their inability to withstand bending through angles of a few degrees, and this was used as a qualitative test. It is general practice to use the elongation characteristics or resistance to fracture on impact as a measure of toughness of a material, but the latter test is difficult to apply to wire and the former is of little value for tungsten, which rarely has total elongation of greater than 1 or 2% at room temperature. A bend test can be of value, but was useless in the range of experimental conditions covered by this work, since, even at the lowest ammonia concentrations used (*c.* 0·02% by volume), the wires were so brittle that bending of more than a few degrees caused fracture. It was possible, however, to measure the tensile strength of the wires on a standard wire-testing machine, the wire being held—to avoid fracture at the grips—at the ends which had been kept cool during heating of the central portion. The loss in strength over that of control specimens, while not entirely a satisfactory measure, served as a valuable guide to the embrittling effect.

Results

Effect of Concentration at Constant Temperature and Time —The tensile strength of the wires was determined after they had been treated in various concentrations of ammonia at 1,200° C. for 10 minutes : 1,200° C. was

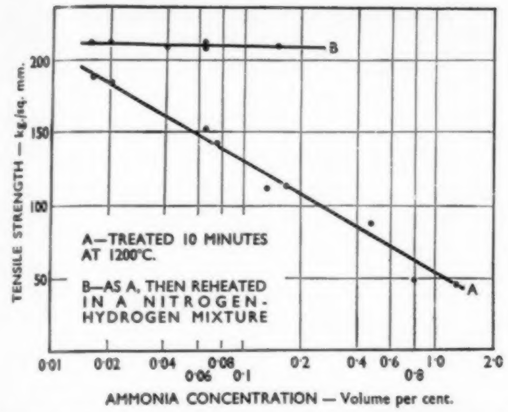

Fig. 1.—Effect of ammonia concentration on tungsten wire heated for 10 minutes at 1,200° C.

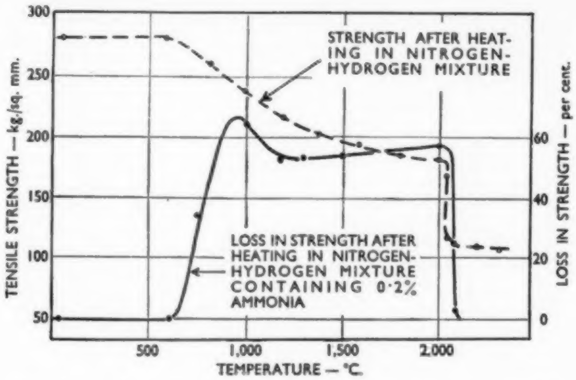

Fig. 2.—Effect of temperature on tungsten wire heated for 10 minutes in 0·2% ammonia.

chosen because preliminary trials had shown that the effect was not very sensitive to small variations about this temperature. The results are plotted in Fig. 1 in terms of loss in strength over wires heated in ammonia-free atmosphere.

When the embrittled wires were retreated at the same temperature, and for the same time, in a nitrogen-hydrogen atmosphere free from ammonia, there was an almost complete recovery of the tensile strength and removal of the brittleness. Very occasionally, small isolated brittle spots remained after this treatment, but the brittleness was confined to portions of the wire less than 1 mm. in length; they occurred at random throughout the whole series of experiments, and were probably due to original defects in the wire.

Effect of Temperature at Constant Concentration and Time.—The effect of the temperature of heating was examined, using a fixed concentration of 0·2% by volume of ammonia and a heating time of 10 minutes. Since the heating itself had the usual softening effects due to recrystallisation and grain growth, it was necessary to allow for the effects of these structural changes. Most commercial tungsten wires of diameters below c. 500μ have a tensile-strength annealing-temperature curve showing two stages of softening.[11] The first stage is associated with primary recrystallisation, which leaves the wire in a ductile state, while secondary recrystallisation, which gives a coarsened structure, leads to an embrittled condition and takes place at temperatures near 2,000° C.

In Fig. 2, the normal softening curve showing these recrystallisation characteristics is given as a broken line, and the effect of the ammonia treatment is shown by the full line in terms of the percentage loss in strength over that of wires treated in ammonia-free atmospheres. The course of this curve suggests that no reaction occurred below c. 600° C. or above c. 2,100° C., and that the maximum effect occurred near 800–1,000° C.

Specimens of wire pre-heated at (a) 2,000° C. and (b) 2,400° C., i.e., characteristic of wire in the states of completed primary and secondary recrystallisation, respectively, were heated in atmospheres containing 0·2% ammonia gas. The tensile results on the specimens pre-heated at 2,000° C. showed that the pre-treatment generally reduced the intensity of the effect, and that the embrittlement occurred over the range of 700–1,600° C. (no tests were made above 1,700° C.), with a maximum effect near 1,000° C.

The effect of pre-treatment at 2,400° C. was to prevent the manifestation of embrittlement by ammonia completely. This may have been due to true absence of ammonia embrittlement, or to the effect having been obscured by the embrittling due to the coarsening process, but certainly there was no further loss in strength on these specimens after ammonia treatment.

Effect of Time at Constant Concentration and Temperature.—A fixed concentration of 0·2% by volume of ammonia and a temperature of 1,200° C. was used to investigate the effect of various times of treatment; two sizes of wire were used, of 250 and 500μ diameter, respectively. The results are plotted in Fig. 3, which shows how the embrittling action increased with time, the effect being more marked with the finer wire.

Active Nitrogen and Hydrogen.—Wires were heated in atmospheres of pure nitrogen and hydrogen, the gases being subjected to a discharge from a Tesla coil. No embrittling effect was observed.

Nature of the Wire Surface.—To ascertain whether the reaction was associated with the clean metal surface itself, or with contaminants there (oxides, graphite, traces of other metals, etc.), wires were polished elec-

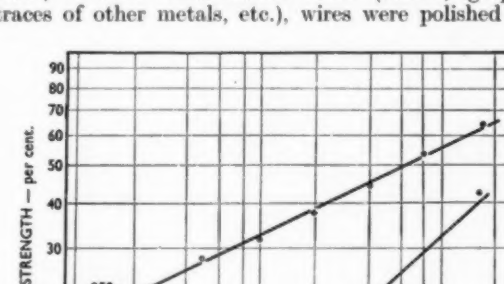

Fig. 3.—Effect of time on tungsten wire heated at 1,200° C. in 0·2% ammonia.

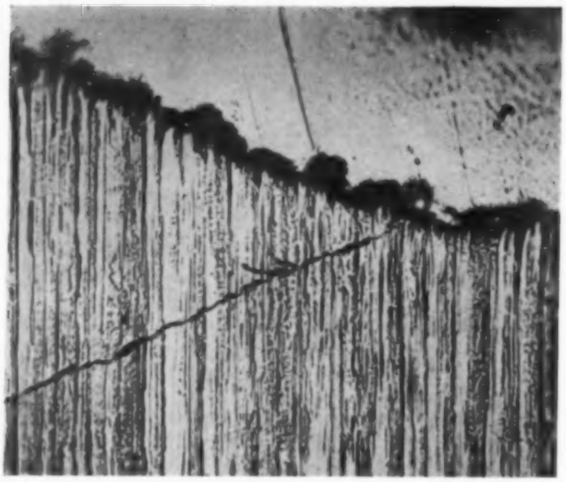

Fig. 4.—**Section through fracture in an embrittled tungsten wire. Etched.** × 1,200

trolytically before treatment in ammonia. The embrittling action was almost completely eliminated, being undetectable in many cases, even with an ammonia concentration of 1%. Occasional spots still occurred, but even then the effect was small. If half the wire to be treated were polished, embrittlement was confined to the unpolished section.

Polished wires were locally oxidised and, after treatment in the presence of ammonia vapour, became brittle at and near the oxidised areas. A control wire treated in the same way, but with the atmosphere free from ammonia, was not embrittled.

Wires treated in 1% ammonia were electrolytically polished and found to be non-brittle. Increasing the concentration to 2% ammonia rendered the wires extremely fragile, but the ductility was restored by electrolytically polishing the surface, removing 5% of the diameter.

X-Ray Examination.—X-ray diffraction examination of embrittled wires showed that no detectable distortion of the tungsten lattice has occurred, but a second phase, at first unidentified, was observed to be present. The lattice of this substance was subsequently found to conform to the values given by Kiessling[10] for the β-nitride of tungsten.

Microscopical Examination.—All the test samples were mounted, sectioned and examined microscopically, using a wide variety of etching reagents, but no indication could be obtained of the presence of any precipitating phase, or, indeed, of any change in the microstructure. Examination of the fractures of embrittled specimens showed that fracture occurred in a transcrystalline manner. Fig. 4 illustrates a typical specimen, and it can be seen that the main fracture and its subsidiary fractures were completely transcrystalline and that there had been no reduction of area (necking) at the point of fracture. An untreated wire usually shows some necking when broken under tension at room temperature.

Discussion and Conclusions

From the above results, it is difficult to arrive at a completely convincing picture of the embrittling action, but the following conclusions seem justified :—

(1) The embrittling action seems to be due to reaction with ammonia itself, rather than its dissociation products, and is associated with the presence of nitride as an insoluble phase.

(2) The effects of time, ammonia concentration, and specimen diameter, make it evident that the reaction is one involving diffusion of some agent into the metal from the surface.

(3) Either the reaction is initiated (or catalysed) by oxide on the surface, or the extent of the effect is strongly dependent upon surface area—which would be decreased by polishing and increased by hydrogen reduction of oxidised areas at the surface.

(4) The effect is not apparent with specimens which have undergone coarsening by secondary recrystallisation. This fact might support a supposition that penetration is facilitated by the presence of numerous grain boundaries, but it may simply be a result of the high temperature heating, which cleans up the surface and tends to smoothen it. Further, of course, the embrittlement consequent upon the coarsening itself may predominate.

(5) Since the embrittlement is removed by dissolving off a disproportionately small quantity from the surface, this defective surface material must act as a source of ready initiation of cracks, which then propagate through the unaffected interior of the specimen. Tungsten is, in fact, very sensitive to the notch effect, and the deformability of wires is drastically reduced by relatively slight surface defects.

From a practical point of view, it is imperative that traces of ammonia should be excluded from atmospheres in which tungsten is to be heated, although the effects can be mitigated by certain pre- or post-treatments.

Acknowledgment

The author wishes to thank Mr. J. A. M. van Moll and the Directors of Mullard, Ltd., for permission to publish this paper.

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New Crofts Branches

Crofts (Engineers), Ltd., the power transmission engineers, have opened two new Midlands branch offices. They are situated at 47–49, Derngate Chambers, Northampton (Tel. : Northampton 3560), where Mr. A. T. Gleave is Branch Manager, and at Room 4a, Majestic Chambers, Campbell Place, Stoke-on-Trent (Tel. : Stoke-on-Trent 44930) under Mr. R. Moss.

Change of Address

The Lancashire and North-Western Sales Area Office of Thos. Firth and John Brown, Ltd., hitherto in Manchester, now operates from 180–182, Sefton House, Exchange Buildings, Liverpool, 2 (Tel. : Central 7278–9).

Dud Dudley

An Unrewarded Pioneer

By Eric N. Simons

The rapid depletion of the forests of Britain in the early part of the 17th century led to many attempts to use pit coal for the smelting of iron. The subject of this article, Dud Dudley, is believed to have developed a process whereby this could be achieved, although the product may not have been of the highest quality.

At the start of the 17th century, and perhaps even somewhat earlier, those whose primary concern was the maintenance of the British iron industry began to consider with much earnestness the drawbacks of charcoal as a fuel for smelting, and the possibility that it might be replaceable by some other material. There were increasing restrictions of a legislative character on the industry as a whole, owing to the fact that the rising demand for charcoal was making great inroads on the forests of the country, so causing a shortage of timber, and in consequence raising its price. A wise iron smelter could foresee the time when charcoal would be prohibitive in cost and scarce in supply.

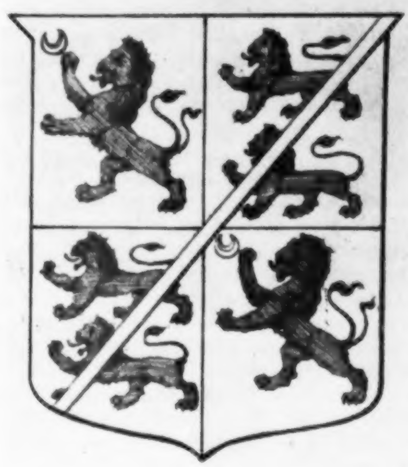
The only possible alternative was pit coal, of which seams were, even in those days, plentiful in the majority of the iron-producing districts. During the reigns of James I and Charles I, efforts to use this coal for smelting iron had been made, but had proved failures. The result was that in many iron-producing districts of Britain, iron works had ceased operations altogether, while elsewhere activities had to be considerably reduced.

Early Attempts at Using Coal for Smelting

In 1607, Robert Chambrell was granted a patent for an attempt to use fossil fuel as a substitute for charcoal, but this led to nothing. Four years later, Simon Sturtevant patented the use of mineral coal for smelting iron, but he was unsuccessful and his patent lapsed. In 1613, John Rovenzon or Rolvenson was also awarded a similar patent, and it has been claimed that he succeeded in doing what Sturtevant had failed to do, but there is no proof of this. There were two other failures, and then, in 1619, a man of the name of Dud Dudley appeared on the scene.

Dudley was a remarkable inventive genius, and there is good reason to believe that he succeeded in making iron with pit coal, the fuel serving for both wrought and cast metal. The story of Dud Dudley is one of the most fascinating in the entire history of metallurgy, and no apology is made for presenting it in full. Some doubt exists as to the genuineness of Dudley's technical success, but this point will be dealt with in due course.

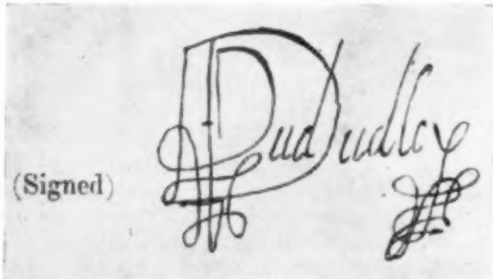
In England at the beginning of the 17th century (1613) there were, according to Dudley himself, 300 charcoal furnaces at work, each turning out 13 tons of iron a week, the total yearly aggregate being 180,000 tons. Of this amount, 112,500 tons were used in making 75,000 tons of bar iron, the rest being used to make cast iron ware. In 1611, if Simon Sturtevant's figures are accurate there were 800 mills in existence in Great Britain for iron manufacture: 200 in Wales; 400 in Surrey, Kent and Sussex; and 200 in other parts of England, Scotland and Ireland.



Dud Dudley's arms.

In 1637, Charles I, by proclamation, ordered the pigs and bars made in England to be marked by his surveyors of iron works, to prevent the sale of bad iron. He also prohibited all export of the metal, except under royal licence. Failure to observe these conditions led to forfeiture of any iron concerned. In 1665, during the reign of Charles II, Dudley, then poor and neglected, published under the title of "Metallum Martis," his version of his efforts to smelt iron with mineral coal. He records that he had no motive, private or political, other than the common weal and the avoidance of destructive inroads upon the wood and timber of the land.

His own father was an ironmaster, and Dudley, when twenty years old, was brought back from Oxford to superintend and manage three of his father's iron works, consisting of a furnace and two forges at Pensnet in Worcestershire. Fresh from college, he was at once faced with the realities of the iron industry. Wood and charcoal were becoming ever scarcer and their cost mounting enormously. All about his furnace were, however, ample supplies of this hitherto unserviceable pit coal. It struck him as ridiculous that this abundance of potential fuel should be unused. He began to experiment, and claims that at his very first attempt he



Dud Dudley's signature.

" made iron to profit with pit coal." A second trial blast was equally satisfactory, the iron produced being of good quality and commercially marketable, but he did not turn out more than 3 tons a week. Overjoyed at his success, he told his father of his experiments, and pressed him to seek a patent from King James. The patent was speedily granted.

Flood Disaster

There were nearly 20,000 smiths of sorts within ten miles of Dudley Castle, and many iron works within that circle had decayed for lack of wood. If Dudley is to be believed, a great flood caused by excessive rain ruined his works and his inventions, as well as the works of many others, and " at a market town, called Sturbridge, *in comitatu Wigorniae*, although the author sent with speed to preserve the people from drowning, one resolute man was carried from the bridge there in the day time; and the nether part of the town was so deep in water, that the people had much ado to preserve their lives in the uppermost rooms of their houses." (This even today has a familiar ring !)

His loss, he says, was greeted with joy by those iron-masters whose works had escaped the flood, and who " had often disparaged the author's inventions, because the author sold good iron cheaper than they could afford it, and which induced many of the iron masters to complain unto King James, averring that the iron was not merchantable."

Dudley repaired his works, however, and the King, in response to these complaints, ordered him to send all sorts of bar iron to the Tower of London " to be tried by all artists, who did very well approve of the iron; and the said Parkhouse had a fowling gun there, made of pit coal iron, with his name gilt upon the gun, which gun was taken from him by Colonel Leveson, Governor of Dudley Castle, and never restored."

The success of these trials is said to have silenced Dudley's enemies for a time. In the Parliament of that day, legislation was being drawn up with a view to nullifying monopolies. Some of Dudley's competitors tried to get his pit coal process declared a monopoly, but with the help of Lord Dudley, this attempt was defeated, and the inventor went on making his iron and selling it, he tells us, at £12 a ton. The Statute of Monopolies specifically excluded from its provisions " the melting of iron ewer, and of making the same into cast-works or barres, with seacoales or pitcoales." This Dudley regarded as a great victory for himself.

He also made brewing cisterns, pots, mortars, and other cast iron goods " better and cheaper than any yet were made in these nations with charcoal." Later, he says, he was " ousted out of his works and inventions before mentioned, by the iron masters and others, wrongfully," but he does not say how. Nevertheless, " unwilling that his inventions (having undergone such charge and pains therein) should fall to the ground and be buried with him," he set up again at Himley furnace in Staffordshire. Here he made iron from pit coal, but not possessing a forge, he had to sell his iron to the iron-masters, who " did him much prejudice, not only in detaining his stock, but also disparaging the iron—Himley furnace being rented out unto charcoal iron masters."

Dudley's sorrows were far from being ended. He built a large new furnace at Hascobridge in the parish of Sedgely and County of Stafford, with larger bellows than any other furnace possessed. Here he produced 7 tons of iron a week, the largest quantity of pit coal iron yet made in Great Britain. He discovered new coal seams, 10 yds. thick, near the furnace, with iron under them. He had no sooner got these coal works in order than he was thrown out of them by force, and the bellows of his furnace were cut to pieces by rioters, instigated by his rivals.

What with lawsuits and riots, Dudley now found himself " wearied and disabled to prosecute his art and invention . . . even until the first patent was extinct." He was imprisoned—wrongfully, he asserts—for a debt of several thousand pounds in the Counter in London, yet in the fourteenth year of Charles I's reign, he obtained a new patent. Powerful ironmasters (Sir Philibeard Vernat, a Dutchman, who also obtained a patent, and Captain Whitmore) opposed him, whereupon Dudley and his three partners petitioned the King to the effect that " Sir Philibeard had, three years after he had been ordered at Council board to perfect his invention within two years, made little or no iron, and neither benefited himself nor the Kingdom."

Civil War

The petition was referred to the Attorney-General and the Solicitor-General; but the two opponents did not appear in support of their own patent. Civil war between Parliament and King caused Dudley's partners to abandon the project, and Dudley's own estate was sold. Still undaunted, and with untiring energy, he took two new partners, Walter Sevens, a linen draper of Bristow, and John Stone, a merchant of the same city. New works were started at Bristow " and there we three partners had in stock near 700 l.; but they not only cunningly drew me into bond, entered upon my stock and work, unto this day detained it, but also did unjustly enter staple actions in Bristow, of great value against me, because I was of the King's party, unto the great prejudice of my inventions and proceedings, my patent being then almost extinct; for which, and my stock, I am forced to sue them in Chancery."

While these Chancery proceedings were pending, Cromwell and the Parliament granted a patent to Captain Buck of Hampton Road for making iron with pit coal and sea coal. At great cost works were erected in the Forest of Dean, Cromwell and many of his officers being partners in the enterprise, as well as many " doctors of physic " and merchants. Their experiments failed. Dudley was eventually called in and built a new furnace for Buck and his partner, though as a Colonel of the King's party, he was in great danger from the

Parliament. He told his employers bluntly that they would not succeed, and although they put up nine different works at Bristow, he proved correct.

Another of the partners, Captain Wildman, although a minister—as Dudley pathetically observes—bought Dudley's estate for £200 a year, meaning to wring his secrets from him. Wildman afterwards passed the estate on to two London brokers, who pulled down Dudley's two mansions and sold 500 of his trees. In 1655 these mansions were still unrepaired. Buck's abortive experiments ended in 1656.

Another Captain, John Copley, obtained a fresh patent for making iron with pit coal and sea coal, and set up works at Bristow near the coal works. He tried to blow his bellows mechanically and failed.

Dudley, consulted again, told Copley he would fail even if he persuaded his bellows to work, but without payment he "did perform the blowing of the said bellows . . . a very forcible and plausible way, that one man may blow them with pleasure" (*sic*), "the space of an hour or two; and this" Copley himself wrote, "I do acknowledge to be performed with a very small charge and without any money paid to him for the said invention."

Copley fulfilled Dudley's prediction and failed, going to Ireland in 1657. Experiments with pit coal then ceased, but in 1660, Dudley, now 61, petitioned Charles II that he might be restored to his place and his obstructed patent revived. He claimed that he had sold his pig iron at £4 a ton with good profit. How great his foresight was may be judged from this passage from his book: "If the coals and iron stone so abounding were made right use of, we need not want iron as we do, for very many measures of iron stone are placed together under the great 10 yds. thickness of coal . . . this coal and iron stone should give the first and last occasion for the invention of making iron with pit coal."

His petition was not granted. Weary, broken in health and spirit, ruined and forsaken, a thwarted genius who lived too soon, he was compelled at last to give up all his attempts, and died in 1684.

Unpleasant Character

While his misfortunes arouse our sympathy, there is another side to the matter. According to Professor T. S. Ashton in "Iron and Steel in the Industrial Revolution," Dudley's personal character was unpleasant. He is said to have delighted in controversy and contention, was always going to law, and was so great a talker that his mother, in her will (he was an illegitimate son of one of the Earls of Dudley), expressly ordered that he should not see her papers, as otherwise many people might be injured by his gossip.

This, however, does not of itself invalidate his claims, but Professor Ashton points out that he (Dudley) never mentioned that he at any time tried to coke the coal, and in view of the feeble blowing apparatus available at that time, it is doubtful, he considers, whether sound iron could have been produced with the raw fuel. (In this connection we must bear in mind that Dudley's bellows were said to be the biggest yet known).

Professor Ashton believes that Dudley did discover a process whereby iron, not necessarily of the highest quality, could be made from mineral fuel, and this undoubtedly ranks him as a great metallurgical genius and one to whom a debt is owed, because even if his own contribution to metallurgical progress was not greatly successful, he undoubtedly focused men's minds on the potential value of pit coal so firmly that the eventual triumph of Abraham Darby was brought nearer, perhaps, than it otherwise would have been.

£650,000 Mill Building Programme at Samuel Fox

INCREASED output of high carbon precision cold rolled strip and the consolidation of stainless steel strip production are the principal objectives of a £650,000 rolling mill programme now in progress at the Stocksbridge works of Samuel Fox & Co., Ltd., a subsidiary of The United Steel Cos., Ltd. The new plant will come into service progressively from July onwards and when the last units have been installed, by mid-1957, it is expected that an additional 30 to 40 men will be needed.

Precision Cold Rolled Strip

Three new mills are being laid down to produce high carbon precision cold rolled strip, which is used in the manufacture of such products as razor blades and clock and watch springs. The existing plant at Stocksbridge rolls the strip in half-ton coils up to 6 in. wide; the new mills will be capable of handling one-ton coils and rolling up to 9 in. wide. On completion of the Brinsworth continuous hot strip mill, at present being laid down by Steel, Peech and Tozer, Rotherham—another member of the United Steel group—the heavier coils of strip will be supplied by the Rotherham works.

Due for commissioning in July, the first of the three new mills is a high-speed, four-high reversing mill, designed by Cold Metal Products of the U.S.A. and built by W. H. A. Robertson & Co., Ltd., Bedford. This unit is of interest in that the backing rolls are driven instead of the work rolls, as on conventional mills; if desired, it can be fitted with tungsten carbide rolls. The mill drive consists of two 100 h.p. electric motors, the coilers on either side of the mill being driven by two 75 h.p. motors. The other two mills are of the conventional four-high reversing type, and are being designed and made by W. H. A. Robertson & Co., Ltd. One mill is driven by a 350 h.p. electric motor with a 70 h.p. motor for each of the two coilers, the other mill being driven by a 200 h.p. motor with 51 h.p. motors for the coilers. All the electrical equipment for these three mills is being supplied by the English Electric Co., Ltd.

Special handling equipment, forming a common system for the three mills, is being provided, with the aim of securing a quick turn-round and thus increased productivity. Additional heat treatment facilities are to be installed, including strand and batch annealing furnaces; there will also be two new multiple slitting machines.

Stainless Steel Strip

In order to consolidate production of stainless steel strip at the Stocksbridge works, two existing four-high

cold mills are being transferred from the company's Rotherfield works at Sheffield, and will be housed in the same building as the new mills. Opportunity is being taken during the transfer to improve the mills, which will operate in tandem on the production of stainless steel strip up to 12 in. wide. Main drives for the two mills are 150 h.p. electric motors, with a 25 h.p. motor driving the coiler at the end of the train.

New Buildings

Dimensions of the new mill building, which is now completed, are 360 ft. long by 66 ft. wide. The building has a high-low roof and generous areas of glazing to permit the best possible natural lighting conditions; it is equipped with a ten-ton overhead crane supplied by Joseph Booth & Bros. Running the full length of the bay is a partitioned-off motor room which, in addition to housing electrical equipment for the new mills, also contains certain equipment for the existing rolling mills in the adjacent cold rolled strip department. A warehouse extension, 45 ft. long by 66 ft. wide, has been erected at one end of the building.

Erection is about to begin on an additional bay, 280 ft. long by 60 ft. wide, to accommodate continuous softening and descaling plant for stainless steel strip; this bay will also be used as a warehouse for stocking stainless steel. Main contractors for the fabrication and erection of the mill building and strip descaling bay are S. Butler & Co., Ltd., the foundations being prepared by Grant, Lyon & Co., Ltd., and the steelwork supplied by Appleby-Frodingham Steel Company—a branch of The United Steel Cos. Ltd.

The Functions of a Professional Institute

Dr. A. D. Merriman's Address to the Institute of Ceramics

AT the Luncheon which followed the First Annual General Meeting of the Institute of Ceramics, Ltd., held at the North Stafford Hotel, Stoke-on-Trent, on Friday, June 1st, 1956, the guests of honour included Dr. A. D. Merriman, G.C., O.B.E. (Secretary and Registrar of the Institution of Metallurgists); Mr. E. W. Brain (President of the British Pottery Manufacturers Federation); The Hon. Josiah Wedgewood; Mr. J. S. Jones (President of the Institute of Clay Technology); and Mr. E. Simpson (a Director of the Brick Development Association, Ltd.).

Following the Luncheon, Dr. Merriman addressed the gathering on "The Functions of a Professional Institute." Referring to the history and influence of ceramics since pre-historic times, he reminded his audience that the application of ceramics had been almost entirely to peaceful pursuits, and that in all ages craftsmen of great skill and of high repute had been attracted by the work. In recent times there had been added the important ingredient of "understanding" to this work. Thus, as he said, a very young technology has come into being. This, like all the other applied sciences, is based on the study of the pure sciences. Ceramics borrows from the pure sciences, but applies the principles to particular classes of problems and in new ways for specialised ends. And as the basis in pure science is always expanding, so are the frontiers of your technology.

In the sixteenth century, one man could be expected to study and to know about all the relevant parts of scientific knowledge. But growth of all sciences has been so rapid that it has been necessary to sectionalise them, and the same is true of the applied sciences. When, in any one of these fields of applied science, the frontiers are more or less clearly defined; when research within the field is established, and literature builds up; when those working in the field develop a new point of view and envisage their own problems; when the technology makes its own contribution to general scientific knowledge; then the applied science is legitimately considered as a specific and distinct branch of knowledge and a technology in its own right. It is then entitled to be heard; it is entitled to recognition and it is entitled to its own professional institute.

The concept of a professional institute is based on the qualifications and ideals which are required of, and which are maintained by, its members, the dignity of character which the members bring to the performance of their duties, and the austerity of their self-imposed ethical standards. The objective should be to promote in every way the interests of, and to maintain and enhance the status and prestige of, workers in your field, and to encourage trained and experienced scientists whose main work and interests lie in technology to become and designate themselves "ceramists."

Recognition

When the professional body comes into being it has certain functions to perform. I have not been able to select these in an order of importance for there is much overlapping, but for convenience I will mention them separately. Firstly I would bring to your notice the importance of what I shall call recognition—recognition of the body, the profession and the members.

This is not only a matter of interest to the body as a whole but to every individual member. It is indeed a matter of concern to every ceramist in the country, who should regard his membership and his support of the Institute as a matter of duty to himself, his colleagues and his profession generally. Those who are well-established in their appointments may wonder what the Institute has to offer, and to those I would say "Your membership is worth much more than your subscription; your association with the Institute is of benefit to both." All such bodies need wise leadership in Council and guidance in Committees, and these are opportunities for service, and service is required of all of us—Lord Halifax is reported as saying that "Service is the rent we pay for the space we occupy on this Earth." Recognition of your technology and of the technological members is the main function of your Institute. It should be directed to ensure that your views collectively or even individually shall command the same consideration as the views of members of other scientific professions. On the appropriate matters your voice should be heard on local and national committees and councils. The Institute should strive for this. Recognition does not come by

resolution. It may take time, but it will come more quickly as the Institute is more widely and loyally supported. Fully qualified persons should be encouraged to call themselves and to be called "ceramists."

Status

Closely linked with the recognition which the Institute must seek is the matter of the *status* of the profession and of the individual members. The distinctive qualification for membership of any professional body must be that combination of academic proficiency and practical experience which ensures that the member has not only studied the appropriate technology but has shown his ability successfully to apply his knowledge, and that he can be trusted for the honesty of his opinions. Once his status is established, it cannot but react favourably upon his appointment, and upon the rewards for his skill, knowledge and understanding. It is a veritable truism that, in the industrial world at least, what has no name does not exist, or at least is ignored. That is why status must be realised, recognised and acknowledged.

Recruitment and Education

Then recruitment to the profession and eventually to the Institute will be simplified. For it is another of the functions of the professional body to attract the right type of recruit into the profession by providing information on matters relating to study and training. Whether this is done by lectures to school-leavers or by the issue of pamphlets it is now clear, after some experience, that the initial effort must be followed by works visits wherever possible. These should avoid showing the work as consisting only of shop-floor routine, but should also indicate the interesting and even exciting scientific background that lacks none of the glamour of the pure sciences usually taught in schools.

The educational aspect of the Institute's work must be regarded as of the greatest importance, for it is in this field that efforts are likely to be most productive. It is the Institute's function to liaise and confer with local authorities in all areas where there is a demand for education in ceramic science. So far as the professional subjects are concerned, the Institute's Education Committee should make itself responsible for drawing up syllabuses. These should be regarded not only as guides to technical colleges, but should set out for all to see the standard that the Institute requires for admission to its membership.

Europe's Growing Energy Requirements

Importance of Coal Stressed in Two O.E.E.C. Reports

THE possible means of meeting Europe's growing energy demands has been the subject of an enquiry by a Commission set up by the Organization of European Economic Co-operation, under the chairmanship of Sir Harold Hartley, President of the World Power Conference. The main task of the Commission was the collection and collation of information covering the whole field of energy in Western Europe, in order to bring out the major problems which have to be faced over the next twenty years. In its report,* published last month, the main issues dealt with are :—

(*i*) the significance of energy as an essential factor both in production and in the standard of living ;

(*ii*) the rapidly increasing consumption of energy in the expanding economy of the post-war world ;

(*iii*) the change in Western Europe from an energy exporting to an energy importing economy and the effects of this on the general economic position ;

(*iv*) forecasts until 1975 of energy requirements and indigenous energy production showing the size of the energy gap that remains to be filled by increased indigenous production, by increased efficiency of utilisation and by imports ;

(*v*) the contribution that may be expected from nuclear energy by 1975 ;

(*vi*) the economic problems that arise from (*iv*) and the urgent need to concentrate efforts on increasing indigenous production of energy and on improving the efficiency of its utilisation ; and

(*vii*) the scope for more co-operation in the field of energy between member countries.

* "Europe's growing needs of energy—How can they be met?" Obtainable from H.M. Stationery Office, price 8s. net.

The Report brings out very strongly the similarities between the United Kingdom's energy problems and those of other Western European countries.

It fully confirms the view of the British Government that coal will be the mainstay of our energy supplies for many years yet. A particularly important point is the Commission's forthright emphasis on the need for "giving coal the outlook of a modernised stable industry, which will attract able young men to join it." The popular view that coal is a dying industry is demolished thoroughly and decisively, and a warning is given against the "popular misconception that nuclear energy will make large contributions in the near future."

Though it does not underestimate the growing part which nuclear energy will play—and the United Kingdom, of course, is a leader in this field—the Commission estimates that this contribution will not exceed a figure of 80 million tons coal equivalent by 1975—less than 8% of total requirements of the O.E.E.C. countries.

Energy Consumption Rising

Consumption of all forms of energy in Europe—equivalent in 1955 to 730 million tons of coal—is rising so rapidly that in 1975 it is likely, according to the report, to reach a figure equivalent to 1,200 million tons of coal. On the basis of present plans, the portion of total requirements which could be covered in 1975 by the production of primary energy would amount to about 750 million tons coal equivalent, excluding nuclear energy developments, as against about 580 million tons coal equivalent in 1955.

The Commission's view is that, unless a special effort is made to promote the indigenous production of primary energy, the remaining gap between energy requirements and indigenous supplies would have to be bridged by imports of coal and oil, the cost of which would amount

in 1975 to about 5,000 million dollars at present prices. This, says the Commission, may present a serious problem for some member countries in its effect on the balance of payments.

The Commission calls for a determined effort among O.E.E.C. countries to develop further their indigenous production of all forms of energy. Additional effort should be devoted to the harnessing of undeveloped sources of hydro-power and to the search for new sources of oil and natural gas. The Commission also attaches special importance to the up-grading of primary into secondary forms of energy, such as gas and electricity. The availability of these secondary forms is essential to a rise in the productivity of many industries, and is also a condition for improved standards of living. Competition should be allowed to play its full role in giving the consumer a maximum choice between the different forms of energy.

The report also draws attention to the large possibilities which still exist for saving energy by economies at every stage, and it also recommends that O.E.E.C. countries should promote the mutual supply of capital needed for the production of energy, as well as the pooling of research and development programmes. It was felt that it would be of mutual benefit to increase co-operation in the fields of exchange of energy, of manpower for the production of energy, and of information and experience. The Commission also recommends the setting up of a new Committee to act as adviser to O.E.E.C.; to examine the Commission's conclusions; and periodically to review Europe's energy position and problems.

The United Kingdom Position

Dealing with the energy situation in the United Kingdom, the report points out that coal represents 99% of the total indigenous output of energy and, despite substantial imports of petroleum, coal still represents 86% of total energy consumption.

The United Kingdom is the largest producer of coal in Western Europe and the third largest in the world. It also leads the European countries in the production of electricity—with 93 TWh* in 1955—and holds third place in the world after the U.S.A. and Russia.

The United Kingdom is now faced with a shortage of coal, and in the immediate future will have to rely on a more extensive use of petroleum and fuel oil to supplement the traditional coal in the production of gas and electricity.

A special drive is also being made to produce nuclear energy supplies as quickly as possible—but despite this ambitious programme it is likely that in the next 20 years the United Kingdom will be obliged to maintain, and even increase, its output of coal, and to make use of substantial quantities of imported petroleum.

The Coal Industry in Europe

Another warning against the belief that the coal industry might decline as a result of the advent of nuclear energy is given in the O.E.E.C. Coal Committee's annual report.† Far from supplanting traditional sources of energy, the report states, nuclear power would immediately have to supplement them in order to face a constantly increasing demand. It would be a dangerous error to think that coal, considered as the least adaptable of traditional fuels, would be reduced to a stop-gap, as such a view might cause producers to restrict or suspend their long-term investment programme and this, in the view of the Coal Committee, could only provoke a future fuel crisis more serious than any which had yet been known.

Dealing with the trend of the coal market during 1955 for the member countries of the O.E.E.C. as a whole, the report states that demand increased by 23 million tons, compared with 1954, but production rose by only 2·5 million tons. This resulted in a reduction of 4·5 million tons in pithead stocks and, at the same time, a substantial rise in imports from the United States, which reached 25 million tons.

The sectors which contributed to increased consumption were principally the iron and steel industry—the great activity of which called for a heavy consumption of coal in coking plants for the manufacture of metallurgical coke—followed by electric power stations and various other industries. The demand by transport and household consumers remained practically stable.

In spite of rising demand, prices remained approximately stable, at least within the Coal and Steel Community. Certain changes which occurred in trade in the O.E.E.C. area were primarily due to reductions in United Kingdom exports as a result of increased demands from the home market.

The Committee think that the demand for coal might increase again in 1956 but at a slower rate than in 1955. In the longer term, the increase in energy requirements was likely to continue, an increased demand for solid fuels being a natural corollary. At the same time new prospects were opened for the coal industry in the chemical treatment of coal and total gasification.

Unfortunately, coal production was far from flexible, and despite all efforts to increase the amount mined there were still difficulties in regard to investment and the recruitment of manpower. A strong flow of imports from the United States was expected for some time, but the prices of United States coal delivered in Europe must take account of Atlantic freight rates, and were thus higher than those of coal of European origin.

International Magazine Award

"Noral News," the works magazine of Northern Aluminium Co., Ltd., has won an Award of Excellence, equivalent to second place in its class, in the 1955 competition for house journals sponsored by the International Council of Industrial Editors, with headquarters in the U.S.A. The award was obtained in competition with journals from several countries, chiefly Britain and America, the entries being judged on editorial balance, writing quality and appearance.

First issued in 1951, "Noral News" is a 28-page quarterly magazine which gives news of the company, its products and the activities of its workpeople. It is issued free to employees in all the works and offices of the company and has a circulation of about 7,500. It is edited by Mr. G. G. Walker of the Publicity Division and is printed by Cheney & Sons Ltd., Banbury.

The present award follows two others obtained in the 1954 and 1955 national house journal competitions organised by the British Association of Industrial Editors.

* TWh = 1 Tera Watt Hour = 1,000 million kWh.
† "The Coal Industry in Europe." Obtainable from H.M. Stationery Office, price 6s. net.

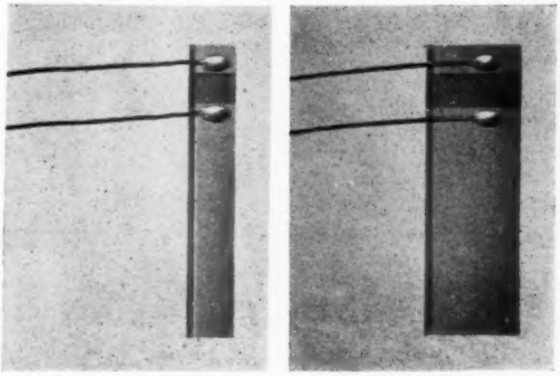
New Barium Titanate Transducers

G.E.C. Accelerometers and Strain Gauges for Vibration Testing

GUIDED missiles, radio valves, motor cars, guns, aeroplanes, steam turbines, ships and certain goods packed in boxes all have at least one thing in common. They are all, at some stage of their lives, subjected to varying forces which may cause them to vibrate. When designing, testing or transporting such equipment, it is often of great value to have some information about the size and frequency of the vibrations taking place. Such information gives the engineer a clearer picture of what is occurring during movement or operation of the object under test, and may help him to modify the design of equipment in the light of the data obtained.

The Research Laboratories of The General Electric Co., Ltd., have now developed miniature barium titanate accelerometers and strain gauges which are already proving extremely useful in the field of vibration testing. They are new devices, the first of their kind to be made available on a large scale in this country, and they have many advantages over older methods of vibration detection and measurement. They are inexpensive, reliable and simple to operate, and their very small size enables them to be used in a variety of circumstances where it would be impossible to accommodate more bulky equipment. In addition, they are extremely sensitive and will work efficiently over a relatively wide range of temperatures and vibration frequencies.

Barium titanate transducers have applications in many branches of engineering, in the manufacture of aircraft, cars, armaments and electronic equipment, as well as in other less obvious fields, such as the transport of packaged goods, where vibration may be experienced.



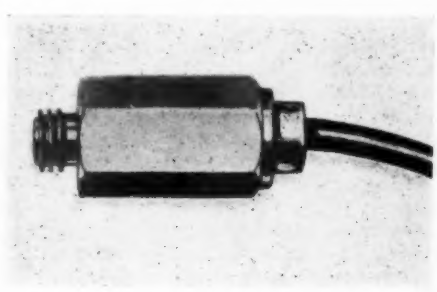
The new G.E.C. vibration strain gauges, which can be used either for detecting or exciting vibrations.

Principles of Operation

Substances of the barium titanate type are classed as ferroelectric materials; that is to say they can, by the application of a large D.C. voltage, be permanently polarised electrically, rather as a ferromagnetic material such as iron can be polarised magnetically. When properly prepared and polarised, barium titanate behaves as a piezoelectric crystal with a very high activity. When such a crystal is compressed, the strain in the crystal gives rise to a minute potential difference across the faces which is proportional to the degree of compression. Conversely, if a potential difference is applied to those faces, the crystal itself will suffer a very small decrease in size.

When subjected to repeated alternating compression and expansion, a piezoelectric crystal will generate an electrical charge proportional to the forces being applied to it. This is what happens in an accelerometer, where the crystal is fixed between a rapidly vibrating surface and an inertia weight. The electrical charge is fed to a detector with a suitably high impedance, such as a cathode follower, and the signal can then be observed on a cathode ray oscilloscope. This method gives the frequency and amplitude of the vibrations. The barium titanate strain gauge is a more simple device, consisting only of a barium titanate crystal with two leads attached. In this case, resonant frequencies can be determined in vibrating mechanical structures, as well as the approximate relative strengths of such vibrations. The great sensitivity of barium titanate transducers depends on the material's very high dielectric constant and piezoelectric activity, which are considerably greater than those of quartz.

Unscreened, (left) and screened versions of the new G.E.C. TypeE barium titanate accelerometer, used for measuring shocks and vibrations.



A comparison between two methods of vibration measurement on a turbine blade. In the foreground is a new G.E.C. barium titanate vibration strain gauge and behind it the much bulkier equipment for the electrodynamic measurement of vibration.

Inset photograph illustrates the extremely small size of the barium titanate vibration strain gauge.

Construction and Operation

Type E Accelerometer

The first of the new devices is the G.E.C. Type E. barium titanate accelerometer, which can detect and measure shocks and vibrations on any object or mechanism over a wide range of operating conditions. It consists of a circular disc of barium titanate, about $\frac{1}{8}$ in. thick and $\frac{3}{8}$ in. in diameter, which is silvered on both sides and sandwiched between two pieces of brass. One piece forms the base and is terminated by a 2 B.A. stud for fixing purposes. The other acts as an inertia weight which converts the vibrations in the equipment to strains in the ceramic. These strains then generate a piezoelectric charge, proportional to the acceleration applied to the base, which is collected by a coaxial screened cable. The signal is taken to a detector with a suitably high impedance, such as a cathode follower, and can be observed on a cathode ray oscillograph. The accelerometer is only $\frac{3}{4}$ in. long, and can easily be attached by its stud to any piece of equipment whose vibration characteristics are to be studied. It is simply and strongly constructed, yet weighs only 11 g. (18 g. with screening can), so that the loading effects on the apparatus under test are minimised. The response of the accelerometer to accelerations of up to 1,000 g.* is linear and its charge sensitivity of about 5 pC./g. gives a voltage output of about 20mV./g. using conventional circuitry. The accelerometer will detect and record frequencies in the range 20 c./s. to 20 kc./s., and between 40 c./s. and 10 kc./s. it is accurate to within $\pm$ 10%. An important design feature of the G.E.C. accelerometer is the construction of the base, which is reduced in thickness just above the mounting stud. This is most effective in reducing spurious signals due to strains in the object under test, and it also reduces the effect of transverse accelerations. The transverse sensitivity of the device is less than 5% of the axial sensitivity, so that the direction of vibration can readily be determined by the position in which the unit is mounted.

Operation of the accelerometer is effective at any temperature between $-$ 50° C. and + 100° C.; if this temperature is exceeded, the piezoelectric properties can be restored by repolarisation. The voltage sensitivity varies with temperature to the extent of only 0·15%/°C. Each unit is individually calibrated at a particular temperature, and is supplied with a 32 in. length of screened cable.

For certain applications it may be preferable to use a screened accelerometer. A screened version of the Type E unit has therefore been designed, with a performance very similar to that of the unscreened type. The aluminium screening-can makes the device somewhat larger, measuring $\frac{5}{8}$ in. by $1\frac{1}{8}$ in. The method of fixing to the test object is equally simple, and the screen may be removed if desired.

Vibration Strain Gauge

The G.E.C. vibration strain gauge was originally developed for measurements of blade vibrations on rotating turbine wheels, but it has many other applications to similar problems. It is two or three thousand times as sensitive to alternating strain as a typical wire resistance strain gauge, but does not respond to steady strain.

The gauge consists of a thin bar of polarised barium titanate with silver electrodes on the two major faces. The electrode on the lower face is extended round one edge on to a small part of the upper face, and the leads are soldered on to the two electrodes on the upper face. The under side of the gauge is attached to the object to be tested by a suitable cement. When the object vibrates, the alternating strains in the plane of the gauge produce an alternating charge on the electrodes, which is fed to a suitably high impedance detector. Unlike the accelerometers, the strain gauges are not supplied calibrated, because they are intended primarily for the determination of the various frequencies at which resonant vibrations occur in mechanical structures, and also of the approximate relative strengths of such vibrations. They will operate in the frequency range 20 c./s. to 50 kc./s.

An important feature of the barium titanate strain gauges, not possessed by other types of gauge, is that they can be used either to detect vibrations or to excite them by the reverse process of applying an alternating voltage to the crystal. This considerably increases the scope of vibration measurements which can be made on mechanical structures.

The strain gauges are made in two standard sizes, measuring $\frac{3}{4} \times \frac{1}{4} \times 0{\cdot}035$ in. and $\frac{3}{4} \times \frac{1}{8} \times 0{\cdot}035$ in., either of which can be used for detection or excitation. The sensitivity is of the order of 0·1 volts output for an alternating displacement of 1 part in 10^6. Like the accelerometer, the strain gauge will operate at temperatures between $-$ 50° C. and + 100° C.; if this temperature is exceeded, the piezoelectric properties can be restored by repolarisation.

* g=gravitational constant.

Rotary Melting Furnaces

Installation in Shell-Moulding Foundry

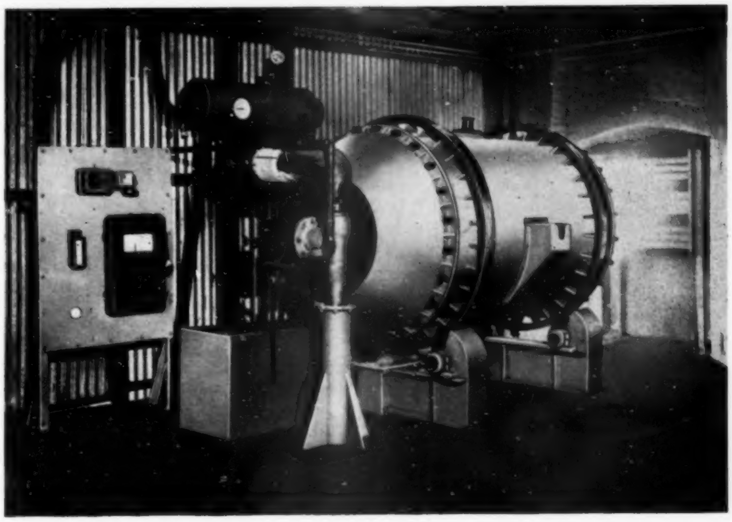


Fig. 1.—Monometer rotary furnace at work in shell-moulding foundry.

ONE of the most important developments in foundry practice of late years has undoubtedly been the introduction of the shell moulding technique, by which castings are produced having a degree of surface finish and "as-cast" tolerances such as would be impracticable by conventional sand-mould methods. It is eminently suitable for the manufacture of light-section castings required to be cast in high-test cast irons, but, as with most processes, shell moulding can only be exploited to its full extent where attention is paid to the installation of suitable ancillary foundry equipment, as shell castings by their very nature require irons of rigidly controlled composition, within a range of different pouring temperatures, at regular periods of the day to suit foundry production requirements.

Founders who have set up shell-moulding departments in their works have come of late to realise the advantages offered by the rotary furnace over the conventional cupola as the melting unit for the production of these castings. The modern rotary furnace melts to consistent analysis, to rigid melting times, and with metal temperatures which can with ease exceed cupola practice when required. Economically also, it can compare favourably with the cupola as a melting unit for this purpose, and many foundries are adopting it as the standard melting unit for this department. A typical furnace installation of this type by the Monometer Manufacturing Co., Ltd., is shown in Fig. 1 producing high-duty cast iron for the shell moulding process in the foundry of J. Hobkirk & Sons, Ltd., at Bedford.

Layout

Such rotary furnaces for iron melting are currently built in cold-charge capacities ranging from ½ ton to 5 tons per charge, and can be fired with a wide range of fuel oils, or with creosote-pitch fuel. In a modern installation of 1 ton capacity, as shown in the illustration and in the general arrangement drawing, Fig. 2, the swinging burner *A* fires into one end of the furnace, the exhaust gases passing over the specially treated tubes of the recuperator *B* before being passed to the chimney. The recuperator unit and flue are on the ground level, and a retractable flame tube *D* carries the products of combustion from the exhaust end of the furnace to the recuperator *B*. The burner normally used is of the low-pressure type, taking both its atomising and combustion air from a single supply fan *E*, and during melting this combustion air is preheated to 350° C. by the recuperator before it is fed to the burner, a system which ensures a very rapid heat pick-up, fuel economy, and high tapping temperatures. Combustion air is under control for pressure, volume, and temperature while it is being delivered to the burner.

An important feature of these installations is the comprehensive instrumentation, which enables accurate control to be exercised over the air-fuel ratio supplied to the burner, and this precise control in turn is reflected in controlled furnace combustion conditions, ensuring consistent metal analyses from consecutive heats. The full instrumentation gives control over air volume, air pressure, air temperature, fuel pressure, fuel volume, and fuel temperature. The furnace is under push-button control for metal removal.

Life of Lining

A modern furnace of the type described is lined with prepared monolithic material, rammed into place around metal formers supplied with the furnace. When used for the production of cast irons, such a lining gives a life in excess of 100 melts, with a total refractory consumption of approximately 50 lb./ton of metal poured. The usual practice is for the furnace installation to be supplied with a spare furnace body in order to avoid any production delays due to furnace re-lining, as when a spare body is available the furnace can melt continuously without any shut-down for repairs, which is in itself a very attractive feature for the shell-moulding foundry.

Physical test results on irons manufactured in the rotary furnace show high values, giving tensile strengths of 23 to 26 tons/sq. in. without alloy additions. The microstructure shows a very much finer flake graphite formation than is the case with untreated cupola-produced cast irons, and even with these high physical

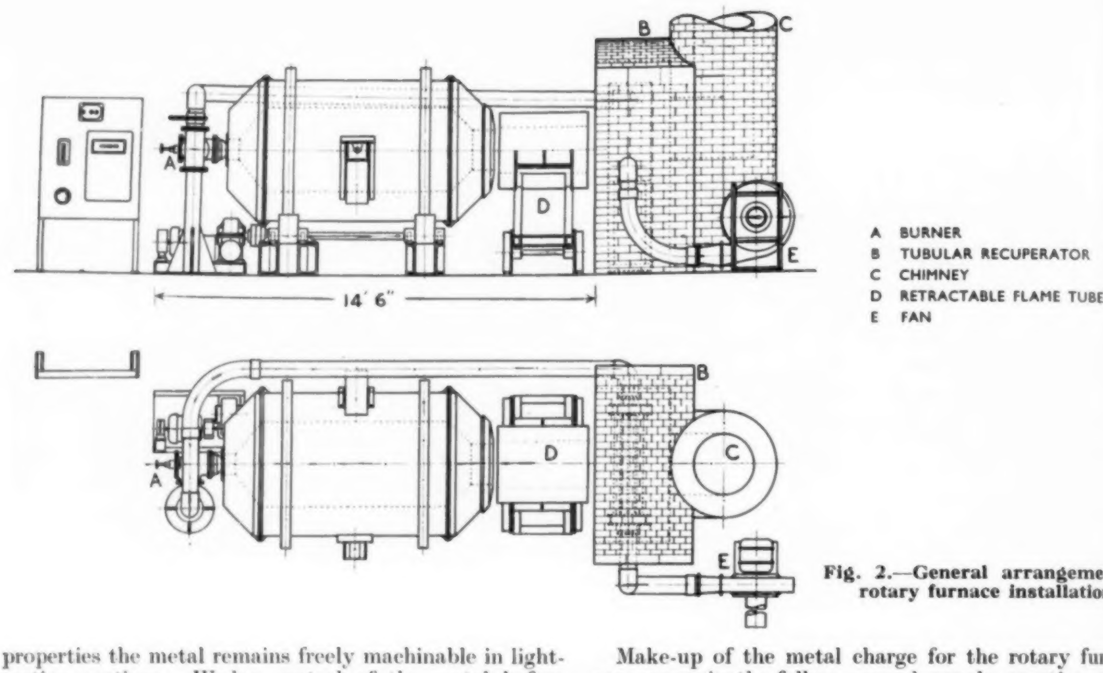


Fig. 2.—General arrangement of rotary furnace installation.

properties the metal remains freely machinable in light-section castings. Wedge control of the metal before pouring, alloying when necessary, metal removal, slag control, etc., are only a few of the operations which are considerably easier to carry out with the rotary furnace, as opposed to the cupola. Metal temperature, in particular, in the rotary furnace is under precise control, and pouring temperatures can be varied at will over a wide range, to suit all casting requirements.

Make-up of the metal charge for the rotary furnace can conveniently follow normal cupola practice, using multiple charges to make the required weight, and pig iron consumption for normal charges will be around 4 to 5 cwt. per ton of metal tapped. The furnace illustrated produces 1 ton of high-duty iron from cold charge to a tapping temperature of 1,500° C. in 1½ hrs., reducing to 1 hr. on continuous melting cycles, with a fuel consumption of 25 to 30 gal./hr.

Aluminium Busbar Alloy

Improved Mechanical Properties

An interesting new aluminium alloy for busbars has recently become available. Given the designation Noral D50S by its makers, Northern Aluminium Co., Ltd., the alloy combines strength with lightness and high current-carrying capacity. The purpose behind the development of the new material has been to arrive at the best possible combination of mechanical and electrical properties, at the minimum cost.

In spite of the emphasis that must be placed on the conductivity of any busbar material, account must be taken as well of the mechanical forces that may arise in any installation. The forces involved as a result of the attraction or repulsion of adjacent conductors can, of course, be considerable—possibly of the order of thousands of pounds per foot run where short-circuit currents are large. The mechanical stresses arising in the busbars depend on the strength of these forces, and on the shape of the busbars and the distance between their supports. It is obvious that an improvement in the mechanical properties of a busbar material will enable supports to be placed more widely, with consequent practical advantages and reductions in installation costs.

Noral D50S has physical properties such that, with the increase in size necessary to compensate for its lower conductivity compared with copper, busbars of this alloy are mechanically, as well as electrically, equivalent to their copper counterparts. Its minimum ultimate tensile strength is 13 tons/sq. in., minimum 0·1% proof stress 10·5 tons/sq. in., and its electrical resistivity 3·133 microhm./cm. at 20° C. (55% I.A.C.S.). Being a fully-heat-treated magnesium-silicide alloy, its conductivity is lower than that of the aluminium of electrical purity hitherto generally used for busbars—the addition of any element to pure aluminium is bound to have this effect. Against this, however, must be counted the fact that its temperature coefficient of resistance (0·00364/° C.) is lower than that of aluminium of electrical purity. This feature does to some extent offset its slightly lower conductivity; busbar ratings are specified in relation to the temperature rise expected above a stated ambient temperature, and for a 40° C. rise on a 30° C. ambient, Noral D50S alloy bars may be rated at 96% of the values given to geometrically-similar bars of electrical-purity aluminium.

The new alloy is available in the form of extruded sections, and could be made available in the form of plate when very large rectangular bars are required. Installations using this material are already in course of erection or being planned.

Ceramic Cutting Tools

High Cutting Speeds Under Suitable Conditions

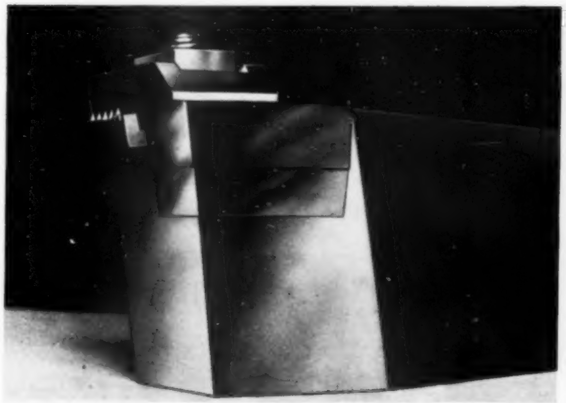
MUCH interest has been shown in the reports from American and Russian sources of spectacular performances of ceramic cutting tools, and Firth Brown Tools, Ltd., of Sheffield, have recently released information on their own work in this field.

Ceramic tools are particularly hard and dense forms of alumina, to which small additions of other substances have been made. In initial cutting tests at Firth Brown Tools, Ltd., tests were carried out on cast iron and mild steel and compared under similar conditions with the hardest grades of hard metal. The results obtained are set out in Table I.

The speeds used in these tests are, of course, much higher than are generally available in industry today, but were necessary in the tests to give measurable tool wear in reasonable time, and to conserve test material. Firth Brown Tools were materially assisted in this work by having installed some years ago a Swift 12·5 in. centre lathe specifically for high speed testing of sintered tool materials.

The ceramic tool material is made up in the form of rectangular pieces, which are mechanically held in a suitable tool holder. It has been found advantageous to provide the tool holder with a hard metal face on which to support the ceramic tip. Tools used so far have had an approach angle of 30° and an included plan angle of 100°. The top rake used has varied from −3° to −8°, little variation in performance occurring over this range. In most cutting tests which have been carried out so far, chip control has been achieved by varying the feed/depth-of-cut ratio rather than by the use of a chip breaker. Early work attempts to use a chip breaker indicated that unless this was critically adjusted, it could have a very adverse effect on tool life.

In addition to tests under the cutting conditions described above, which are at present used as standard tests to evaluate the quality of laboratory-produced batches of material, some initial tests have been carried out on other steels, to try and evaluate the field of usefulness of the new tool material. The results of two such tests are presented in Table II.



Mechanically held ceramic tip with hard metal support.

Further tests have been carried out on the finish machining of austenitic stainless steel, but so far results on this material with ceramic tools have been rather disappointing, due to what appears to be a built up edge. This probably presents a problem on the evaluation of correct tool shape, which will be further investigated. Work is continuing on other steels and on non-ferrous and non-metallic materials.

Firth Brown Tools have found that, under suitable conditions, far higher speeds with much longer tool life can be obtained with the ceramic tools they have pre-

TABLE I.—COMPARATIVE CUTTING TESTS ON STEEL AND CAST IRON USING CERAMIC AND HARD METAL TOOLS.

Material Cut	Speed (ft./min.)	Feed (in.)	Cut (in.)	Duration of Cut (min.)		Flank Wear (mm.)	
				Hard Metal	Ceramic	Hard Metal	Ceramic
Close grained cast iron with a Brinell hardness of 220	1,000	0·010	$\frac{1}{16}$	0·75	38*	0·85–2·02	0·34
0·2% carbon steel with a Brinell hardness of 150	1,500	0·006	$\frac{1}{16}$	7†	45	0·51–1·64	0·34–1·17

* Tool would have cut longer. † Tool cutting very badly after 1 min.

TABLE II.—CUTTING TESTS ON EN.18 AND EN.23 STEELS.

Material Cut	Speed (ft./min.)	Feed (in.)	Cut (in.)	Duration of Cut (min.)	Flank Wear (mm.)
En.18 steel heat treated to a Brinell hardness of 250 ..	1,200	0·004	$\frac{1}{16}$	70	0·25
En.23 steel heat treated to a Brinell hardness of 250 ..	800	0·010	$\frac{1}{8}$	30	0·53

Ceramic tool cutting at 1,500 ft./min. with a feed of 0·006 in. and a depth of cut of $\frac{1}{16}$ in.

pared, than can be obtained with hard metal. While, however, the ceramic material shows to best advantage at high speeds, it will nevertheless give much longer tool life than carbides at intermediate speeds of, say, 300–800 ft./min. The finish obtained with the ceramic tools is extremely good, and much superior even to that obtained with the hardest grade of hard metal.

The brittleness of ceramic tools imposes limitations on their use. Thus, only finish machining operations under good conditions can be carried out, and engaging and disengaging the tool from the work calls for some skill on the part of the operator, if spalling is to be avoided. On the other hand, the tools are not difficult to grind, provided a diamond wheel as used for hard metal tools is used. The tools can best be finish ground on a 400 grit wheel.

It is thought that the further development of ceramics will be complementary to hard metal for high speed machining, but will not have the wide field of application of existing hard metals for general engineering.

There is no evidence to suggest that a ceramic tool will be cheaper than hard metal, the production processes being so involved that the prices may be higher, even though the raw material costs themselves are lower. F.B.T. Research Department is actively continuing development work : (*a*) to assess the field of usefulness of this material ; and (*b*) to continue an improvement of the properties over those already achieved for manufacture for commercial use in the future.

At the present time this work is purely of a development nature and supplies of material cannot be made available.

Institute of Ceramics A.G.M.

THE first Annual General Meeting of the Institute of Ceramics, Ltd., was held at the North Stafford Hotel, Stoke-on-Trent, on Friday, 1st June, 1956, about 80 members being present. In outlining the first year of the Institute's existence, the President, DR. A. T. GREEN, O.B.E., said that incorporation under the Companies Act, 1948, had taken place on 6th May, 1955, and the first full Council Meeting and the first General Meeting had been held on 16th June. On that occasion, the first important step in connection with the election of members had been taken by deciding that the Council, acting in Committee, should itself constitute the Nominations and Examinations Committee. Shortly after that date, applications for admission to the three grades of membership had been received, and there had been a steady flow ever since. Consequently, the work of the Institute had consisted almost entirely of interviewing and assessing applicants who had applied on the basis of external qualifications, and the brunt of this work had been borne by the Interviewing Sub-Committee, to whom the Institute was considerably indebted.

So far, membership was as follows : Fellows, 164 ; Associates, 39 ; and Licentiates, 34. Applications had also been received for admission by examination, but it had not been practicable to work out in detail the necessary syllabus and machinery. But the Council, realising that it was both desirable and necessary to be able to test candidates by examination, had decided to institute for the time being a system of examination by thesis, the candidate of adequate background and training being requested to submit the titles of up to three subjects upon which he would be prepared to write. From these, provided that at least one subject acceptable to the Council was included, a title would be chosen. If, however, none of the titles submitted were considered suitable, the Council would set the candidate a subject ; after the written thesis had been received and considered, the candidate would be orally examined on it. The final result would depend on the candidate's satisfying the examiners in both his written and oral work.

In order to assist the Institute in its early days, donations had been received from : The British Ceramic Society ; The British Pottery Manufacturers' Federation ; The National Federation of Clay Industries ; The Institute of Clay Technology ; The Refractories Association of Great Britain ; and The HON. JOSIAH WEDGWOOD. Since the end of the financial year (31st December, 1955), further donations had been received from : The Brick Development Association, Ltd. ; The Institute of Clayworkers ; and H. J. PLANT, ESQ., J.P., Hon.F.I. Ceram. To all who had given their support to the Institute in this practical way the President expressed the thanks of the Council.

Financial Position

The Hon. Treasurer, MR. L. BULLIN, then presented the Statement of Accounts and Balance Sheet for the year ended 31st December, 1955, which he said he considered was reasonably satisfactory : subscriptions had amounted to £477, entrance fees to £183, making a total subscription income of £660, but he pointed out that it was unlikely that the income from entrance fees would be so high in succeeding years. Moreover, without the generous financial backing of the various guarantors, the Institute could not have been founded—the £730 received had more than covered the legal expenses of incorporation. The running expenses for the seven months had amounted to about £530, leaving the Institute with a credit balance of £290.

MR. BULLIN also referred to the agreement that had been arrived at with the British Ceramic Society, whereby the services of MR. G. H. STEWART were shared, as also was the clerical assistance and office accommodation at Federation House. By this means, it would be possible to keep overhead expenses within reasonable limits.

Election of Officers

Following the examination of the ballot papers by the scrutineers, the results of the Election of Officers and Members of Council for 1956–57, were announced :—

President : DR. A. T. GREEN, O.B.E.

Hon. Treasurer : MR. L. BULLIN.

Vice-Presidents : DR. W. L. GERMAN, MR. G. N. HODSON, M.B.E., J.P., DR. A. L. ROBERTS.

Members of Council : MR. T. G. W. BOXALL, MR. B. BUTTERWORTH, DR. J. H. CHESTERS, MR. F. H. CLEWS, MR. R. S. C. COPELAND, MR. T. C. EDWARDS, MR. J. S. JONES, MR. R. A. KIRBY, J.P., DR. H. H. MACEY, MR. J. H. PLANT, DR. G. R. RIGBY, MR. P. B. ROBINSON, T.D., MR. A. J. C. WATTS, DR. J. WHITE, MR. N. WILSON.

NEWS AND ANNOUNCEMENTS

Welded Pressure Vessel Steels Symposium

A one-day conference on the application of low-alloy steels for welded pressure vessels, organised by the West of Scotland Iron and Steel Institute, will be held on Friday, 19th October, 1956, at 39, Elmbank Crescent Glasgow, C.2, commencing at 10 a.m. The morning session will be devoted to the presentation of the following papers :—

- "Development of Mn-Cr-Mo-V High Tensile Steel for Pressure Vessels," by W. Barr and I. M. Mackenzie (Great Britain).
- "Important Considerations for the Welding of Chromium-Molybdenum Steels," by O. R. Carpenter (United States).
- "Welding of Large Pressure Vessels in Low-Alloy Steels," by Dr. H. Harris (Great Britain).
- "A Low-Alloy Quenched and Tempered Steel for Pressure Vessels," by J. M. Hodge and L. C. Bibber (United States).
- "Some Principles of Design for Pressure Vessels and Boilers made of Low-Alloy Steel," by P. W. Kerkhof (Holland).
- "Properties of Fortiweld Steel," by Dr. L. Reeve (Great Britain).

Some 2½ hours will be available for discussion at the afternoon session, which commences at 2.15 p.m.

The conference is open to all who are interested, on payment of a registration fee of £1 15s., which covers the provision of a set of preprints of the papers. A full report of the conference will be published in the Journal of the West of Scotland Iron and Steel Institute and will also be available for purchase as a reprint.

Further particulars may be obtained from the Secretary of the West of Scotland Iron and Steel Institute, 39, Elmbank Crescent, Glasgow, C.2.

A Conference on the Functions and Education of Welding Engineers

The growth of welding in industry has been extremely rapid but this has not been coupled with a similar growth in the facilities for technical training. The time now seems appropriate to examine the requirements of industry as regards specialised training in welding technology, to survey the present arrangements both in this country and abroad for providing training, and to agree upon ways and means of improving education and training in higher welding technology within the system of technical education in this country. To this end a Conference on the Function and Education of Welding Engineers is being organised by the Institute of Welding for the benefit of technical management, educationalists, personnel officers and welding engineers. The Conference will be held at Ashorne Hill, Leamington Spa, from 16th to 18th September, 1956.

Members will assemble at Ashorne Hill on Sunday, 16th September, and the main work of the Conference will begin at 9.30 a.m. on the following morning, when papers on "The Functions of the Welding Engineer in Industry" will be discussed. In the afternoon, the topic for discussion will be "Welding in Higher Technical Education—The Present Position." The sessions on Tuesday morning and afternoon will be concerned, respectively, with "The Education of Welding Engineers in Other Countries," and "Some Proposals for Improving Higher Education in Welding." All the papers to be presented at the Conference are being printed in the May, June, July and September issues of the *British Welding Journal*.

Further particulars and enrolment forms may be obtained from the Secretary, The Institute of Welding, 2, Buckingham Palace Gardens, Buckingham Palace Road, London, S.W.1. Applications for enrolment should reach the Secretary before 3rd August, 1956.

Export of Copper Goods

There is a control over the export of certain copper goods to ensure that scrap metal is retained in the United Kingdom. In this connection the Board of Trade announce that the Open General Licence which authorises the export of certain copper goods valued at £330 per ton, and of certain copper alloy goods valued at £220 per ton, is being revoked and replaced by a new Open General Licence, which raises the value limit for copper goods to £350 per ton and that for copper alloy goods to £240 per ton.

This licence came into operation on June 15th, 1956, and individual licences are now required where the relevant values are £350 and £240 or less. The copper and copper alloy goods are those specified in Group 6(2) of the First Schedule to the Export of Goods (Control) (Consolidation) Order, 1955. This Open General Licence does not apply to exports to China, Macao, North Viet Nam or Tibet.

Enquiries regarding this licence should be made to the Export Licensing Branch, Atlantic House, Holborn Viaduct, London, E.C.1. (Tel.: City 5733). Copies of the Open General Licence (price 2d. each, by post, 3½d. each) may be obtained from H.M. Stationery Office, Kingsway, London, W.C.2, and branches.

Steel Experts Visit Ural Plants

As a result of arrangments made in the Steel Committee of the Economic Commission for Europe, Soviet steel experts visited the United Kingdom in December, 1954, and in return British experts visited the Soviet Union in September, 1955. Both the United Kingdom and the Soviet Union subscribed at the two most recent Plenary Sessions of the Economic Commission for Europe to resolutions which stressed the benefits of reciprocal exchanges of production experience, and it has now been agreed that a further exchange of visits by steel experts should take place in 1956.

The United Kingdom steel industry therefore invited a Soviet steel delegation to visit this country in the autumn, and accepted the invitation of the Ministry of Ferrous Metallurgy to send a delegation at the end of June to visit plants in the Ural region. The delegation, which left London to fly to Moscow on June 24th, was led by Dr. T. P. Colclough, C.B.E., Technical Adviser to the British Iron and Steel Federation. The other members were: Mr. K. C. Barraclough (Research Officer, Thos. Firth and John Brown, Ltd.); Dr. C. Booth (Assistant Managing Director, Steetley Magnesite Co., Ltd.); Mr. W. O. Campbell Adamson (Assistant General Manager, Redbourn Works, Richard

Thomas & Baldwins, Ltd.); Mr. T. Dennison (Head of Technical and Development Dept., B.I.S.F.); Mr. F. Kennedy (Chief Heat and Fuel Engineer, Dorman Long (Steel), Ltd.); Mr. J. A. Kilby (Chief Mechanical Engineer to Colvilles Group); Mr. A. H. Meadley (Chief Engineer, Iron and Steel Development, Corby, Stewarts & Lloyds, Ltd.); Mr. R. Sewell (Information Officer, United Steel Companies Research and Development Dept.—to act as interpreter); Mr. E. Vose (Head of Iron-Making Division of British Iron and Steel Research Association); and Mr. R. Wilcock (Works Metallurgist, Samuel Fox & Co., Ltd.).

Among the plants seen in the fourteen days' visit were the Magnitorgorsk Metallurgical Combine and the Novo-Tagilsk Metallurgical Works—two of the largest integrated plants in the Soviet Union—the Chelyabinsk Metallurgical Combine, producing special steels, the Chelyabinsk Tube Works, a refractory plant at Nishniy Tagil, and the Ural Research Institute of Ferrous Metals at Sverdlovsk.

Export of Aluminium Goods

The export of aluminium scrap is prohibited, but exports have been made to the Continent of goods described as aluminium semi-manufactures at prices far below those ruling in the United Kingdom. Foreign import returns support the only explanation of this discrepancy, namely, that scrap has been exported declared as semi-manufactures. A minimum price for exports of certain aluminium and aluminium alloy goods has been reimposed after consultation with the Aluminium Industry Council.

In addition, the export of certain aluminium and aluminium alloy goods to any destination is now subject to licence. The relevant Amendment Order is the Export of Goods (Control) (Amendment No. 6) Order, 1956 (S.I. 1956/789). The Board of Trade also announces the issue of an Open General Licence which authorises the export of certain goods of aluminium or aluminium alloy valued at more than £240/ton. This licence does not apply to exports to China, Macao, North Viet Nam or Tibet.

Enquiries regarding the Amendment Order and the Open General Licence should be made to Export Licensing Branch, Atlantic House, Holborn Viaduct, London, E.C.1. (Tel.: City 5733). Copies of the Amendment Order (price 2d., by post 3½d.) and of the Open General Licence (price 2d., by post 3½d.) may be obtained from H.M. Stationery Office, Kingsway, London, W.C.2, and branches.

Co-ordination of B.C.I.R.A. and N.I.F.E.S. Activities

An exchange of views has recently taken place between the British Cast Iron Research Association and the National Industrial Fuel Efficiency Service to ensure that proper co-ordination exists in the technical advice both organizations offer to the ironfounding industry. It has been agreed that the two bodies can co-operate to the advantage of the industry.

The Research Association is, of course, the authority to which the ironfounding industry looks for research, development and technical advice over the whole range of its processes. As is apparent from its title, the National Industrial Fuel Efficiency Service is concerned with fuel problems only. It is not engaged in research, but is concerned to improve the fuel practice of all sections of British industry. Collaboration between the two organisations should, therefore, combine the special knowledge of B.C.I.R.A. scientists and the broad experience of N.I.F.E.S. engineers.

Through its liaison services, the B.C.I.R.A. has been successfully advising ironfounders on specific problems which are encountered in their own works. The field work on fuel that the Research Association undertakes is mainly confined to melting and heat-treatment processes, and is necessarily, therefore, smaller than that carried out by N.I.F.E.S., who have an engineering staff organised on an area basis from offices in 15 cities in Great Britain and Northern Ireland. The B.C.I.R.A. will, therefore, recommend ironfounders who are concerned about the use of fuel for space-heating or steam-raising to consult N.I.F.E.S., who will in turn refer to the Research Association any problems that fall outside its experience.

In order to make this collaboration effective, a party of N.I.F.E.S. engineers who have specialised on high temperature processes recently visited the Research Association's laboratories at Alvechurch. Further conferences between the staff of the two organisations will be held in the future.

The Howitt Committee Report

The Government have accepted the Report* of the Committee which has considered the Crown's powers over the use of technical information needed for fulfilling defence contracts. The Committee, which was appointed last November by the President of the Board of Trade, consisted of Sir Harold Howitt, Sir Robert Barlow and Mr. John Megaw, Q.C. They recommended that the Crown should be given, subject to special safeguards, permanent powers enabling Government Departments to authorise the use (but not the disclosure) of technical information when necessary for defence contracts, and for this purpose to override agreements to the contrary. The essential differences between these permanent powers and the wider Emergency Powers† which they will replace are that they are limited to production for defence (including civil defence): and that they provide for a period of three months for re-negotiation of agreements; for compensation; and, on certain matters, including the amount of compensation, for appeal to the High Court. Only Ministers and senior officials will exercise the powers. There is no right to authorise disclosure (as distinct from use) of the information. These provisions are designed to safeguard industrial interests. Legislation will be necessary to give effect to the recommendations.

The Direction of Research Establishments

The acute shortage of scientists makes good management of research laboratories of particular importance today. The problem of how to choose a research project, of when and how to stop research, and the proper use of research staff and facilities are matters to which considerable attention is being directed and the National Physical

* Published as Command Paper No. 9788, price 1s. (by post 1s. 2d.), obtainable from H.M. Stationery Office, Kingsway, London, W.C.2, and branches.
† The Defence (Patents, Trade Marks, etc.) Regulations, 1941.

Laboratory is to provide a focus for all this thought at an International Symposium on " The Direction of Research Establishments " to be held next September.

The Symposium will be held at the N.P.L. from September 26th to 28th, and will be restricted to directors of research and research administrators; many will be coming from Europe, the Commonwealth and the U.S.A. The opening speakers on the selection of the research project will be Professor J. D. Bernal, F.R.S., and Dr. Willis Jackson, F.R.S. Major discussions will also be held on developing the creativity of scientists, and administrative controls on their work. Three parallel groups will discuss methods of internal organisation, staffing and communication problems, and a final session will be addressed by Mr. A. H. Wilson, F.R.S., on the methods by which the work of a laboratory can be assessed.

Among the chairmen of the main sessions will be Dr. H. W. Melville, F.R.S., the new secretary of the D.S.I.R., and Dr. G. B. B. M. Sutherland, F.R.S., who takes up his post as director of the N.P.L. early in September. Group chairmen are to be Dr. J. Bronowski, of the National Coal Board, Dr. D. Hill, Director of the Shirley Institute, and Dr. A. King, Head of the Intelligence Division, D.S.I.R.

This is believed to be the first international conference on science administration held in this country at which representatives of industry and government organisations will be able to exchange their experiences.

Management Education

In order to meet persistent demand from industry, the Department of Industrial Engineering of the Loughborough College of Technology announces the inauguration, in September next, of a Post-graduate Course of one year's duration in Industrial Engineering. In addition to a wide range of advanced management topics, there are included such special subjects as "Nuclear Energy," "Automation" and "Men and Machines." The first two are approached from the technological viewpoint, whilst the latter is concerned with the physiological effects of noise, fumes, dust, vibration, and so on. The course is suitable for senior personnel from all types and branches of industry.

The three types of courses in Work Study held in the Department are well established and in great demand, whilst the five-year sandwich course in Production Engineering and Industrial Administration, which is attended by the apprentices of many nationally known companies, enters its third year in September.

Particulars of all the courses may be obtained from the Head of the Department of Industrial Engineering, Loughborough College of Technology, Loughborough, Leicestershire. (Tel.: Loughborough 4428.)

Pictures of an Industry

The Inco-Mond Group of companies—The International Nickel Company of Canada, Ltd., The International Nickel Company, Inc., The Mond Nickel Co., Ltd., and Henry Wiggin & Co., Ltd.—are responsible for 63% of the free world's production of nickel, in addition to thirteen other elements, including a vast production of copper and platinum metals. The chief product of this major metallurgical industry, nickel, plays a vital part in nearly every aspect of industry, and in the form of specialised nickel alloys has done much to increase the amenities of modern life. This international organisation owns mines in Ontario, Canada and plants, refineries and research establishments in Canada, the U.S.A., and Great Britain, in addition to information bureaux in the capitals of Europe, in India, Australia and South Africa.

It had long been felt that the importance of the company's operations warranted some permanent record of their activities and a distinguished artist, Terence Cuneo, was commissioned to travel on two continents in an endeavour to capture something of the drama and colour of the industry. The Mond Nickel Company recently presented these graphic paintings of the mining and processing of nickel and its companion metals in an exhibition at Grosvenor House, Park Lane, London, W.1, in an endeavour to bring to a wider public the romance of nickel. The exhibition will also be presented at the Central Art Galleries in New York from September 11th–21st, and at the Chateau Laurier, Ottawa, from October 3rd–6th, and subsequently, in Sudbury, Ontario, Canada.

Light Alloy Conveyor Buckets for Coal and Coke

Long-term economies in coke and coal handling are suggested by the results of a two-year test of special aluminium-manganese alloy conveyor buckets designed and fabricated by the TI subsidiaries, TI Aluminium, Ltd., and the New Conveyor Co., Ltd., and installed at the Windsor Street (Birmingham) gas works with the co-operation of the West Midland Gas Board.

The corrosive and abrasive effects of handling coke, and almost equally coal, it had been found, were such that the effective life of steel buckets on the conveyors was less than three years. After a period of continuous use for more than two years, the experimental aluminium alloy buckets remained virtually as new, with no signs of corrosion or holing, and little denting. The main reason is that the steel buckets rust on contact with the damp coke and coal, and this layer of rust is progressively removed when handling the coke. In consequence, the surface of the bucket constantly becomes thinner to a point where it easily perforates. As aluminium does not rust, there is no removal of metal.

The frames attaching the buckets to the conveyer were of malleable iron, and were not replaced when the aluminium alloy buckets were installed. The two metals were insulated from each other by a simple zinc chromate coating, which proved completely adequate in preventing any bi-metallic corrosion.

The adoption of aluminium alloy buckets, because they are lighter, also reduces the amount of power required to operate conveyor belts.

H.F. Iron Melting

Birlec, Ltd., announce that an important contract has been placed with them by Wellworthy, Ltd., Lymington, Hants., for a high frequency melting furnace installation. Comprising one 1,250 kW. motor-alternator set and two furnaces, each of 3 tons capacity, the installation is of particular interest since, in the United Kingdom at least, it will be the largest high frequency unit to be commissioned specifically for the production of grey and alloy iron castings. To ensure a constant supply of hot metal to the casting shop, a 10 ton Birlec gas-fired holding furnace will also be provided.

RECENT DEVELOPMENTS
MATERIALS : PROCESSES : EQUIPMENT

Bright Cadmium Plating

A NEW bright cadmium plating process (No. 53), based on a development recently tested in the United States by Efco-Udylite, is now available in this country from the Electro-Chemical Engineering Co., Ltd. This process has outstanding advantages which include a wider bright plating range, higher current density of operation, freedom from pitting, minimum sludging and simplicity of control. The type of article to be plated governs to a large extent the composition of the solution used. Thus, process No. 53A is for general use, including work plated in manually operated tanks, semi-automatic or fully automatic machines; process No. 53B is used for deeply recessed articles, where complete coverage with cadmium is important; and process No. 53C is used for barrel plating.

The bath constituents consist of cadmium, of a content which varies with the items to be plated; sodium cyanide, which provides solution conductivity and anode corrosion; caustic soda, which is formed when cadmium oxide and sodium cyanide are dissolved in water; sodium carbonate, which is unavoidably formed by decomposition of the sodium cyanide; and Brycad brightener No. 53, which is a liquid added to the plating solution in quantities depending on the cadmium content of the bath and the brightness required.

The operating range is 75°–95° F. Higher temperatures permit higher current densities, but are harmful to the brightness and throwing power. Lower temperatures decrease the conductivity, thereby lowering the current density considerably. As with any other plating process, adequate cleaning, pickling and rinsing facilities are necessary to ensure that the deposit possesses a satisfactory degree of adhesion. Cadmium plate should not, of course, be put on articles to be used with food and drink.

In those cases where it is required to convert an existing cadmium plating solution to an Efco-Udylite Brycad No. 53 bath, a sample of the solution concerned can be sent by some suitable means to the Woking Laboratories for analysis and trial conversion. Most solutions can be converted by the addition of suitable materials to give satisfactory results.

Electrochemical Engineering Co., Ltd., Forsyth Road, Sheerwater Trading Estate, Woking, Surrey.

Sections of steel curtain rod before and after plating in a Brycad 53 bath.

Model 58 Consotrol Controller

THE Foxboro M/58 Consotrol Controller is an important unit in the well-known Consotrol range of instruments. This range, including recorders, indicators and controllers, is designed primarily for use in graphic panels. The M/58 is a new development in pneumatic precision control instruments, and employs a floating disc, force balance system which gives exceptional stability and sensitivity. This simplified balancing system has measurement, proportional set and integral bellows acting on a single disc. There is only one nozzle and one moving part, and the system operates with the minimum of friction and dead space, and responds instantly and accurately to the smallest change in measurement and set pressures. The system is unaffected by ambient temperature changes, and its simplicity of design has resulted in a highly compact instrument. (Overall size $5\frac{3}{16} \times 4\frac{3}{4} \times 3\frac{3}{8}$ in.)

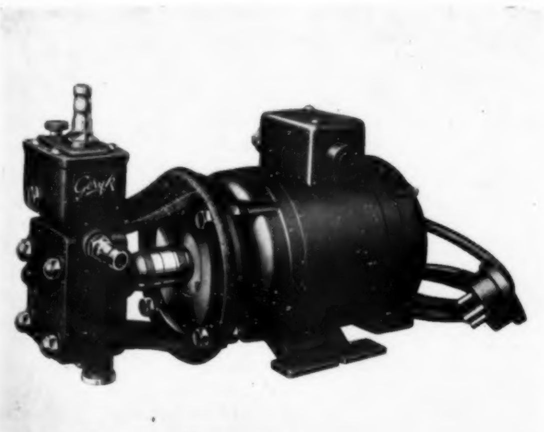
The Foxboro Model 58 Consotrol controller

The M/58 provides four control actions: proportional; proportional-derivative; proportional-reset; and proportional-reset-derivative; and an important feature of the instrument is the ease with which it may be converted from one to the other, in the field, by simply adding or subtracting standard components, and without disturbing air piping or the remote manual operation of the process. Furthermore, the control actions may be adjusted easily and quickly over very wide ranges, the proportional band having a range of setting from zero to infinity, while the reset and derivative time is adjustable over a range of 1,000 to 1.

Foxboro-Yoxall, Ltd., Lombard Road, Merton, London, S.W.19.

Compact Vacuum Pump

To meet the demand for an efficient portable vacuum pump which can be moved around easily in a laboratory or workshop the Pulsometer Engineering Company have developed their 1-in. F.R. Rotary Vacuum and Pressure Pump. The weight of the complete unit with motor is only 33 lb. It is completely free of vibration and quiet



1 in. Pulsometer F.R. pump

in running, permitting it to be used on a bench or table without bolting down ,and is very simple to clean and re-assemble.

The close-grained cast iron cylinder has mounted in it an eccentric rotor of smaller diameter, running in line contact with the top of the ground horizontal bore. The rotor carries two sliding vanes, running in a closely-toleranced slot, which sweep out the air space in the cylinder of the rotor. The top of the cylinder casting is extended to form an oil well, which seals the rotary-disc type discharge valve, and feeds oil to the cylinder bore for lubrication purposes.

To prevent air leakage to the pump along the driving shaft, a special rotary seal is used which, beside being perfectly airtight under working conditions, reduces the power absorbed and does not score and wear the shaft. This seal consists of a hardened ground and lapped steel cup, which is pressed by means of a spring against a prepared rubbing face on the cylinder cover. Interposed between the spring and cup is a square sectioned rubber ring backed by a steel follower ring, the rubber ring being a tight fit on the shaft and in the cup. Driven by

Attachment for Nelson stud welding Tool

the fit of the rubber ring on the shaft, the whole assembly rotates with the lapped face of the cup bearing on the stationary front cover. To complete the seal, the entire space is filled with oil fed from the main system. The action of the oil on the rubber ring makes it stick firmly to the shaft and rotary seal cup, and this adhesion is so strong that firm pressure is needed to dismantle the rotary seal from the shaft after it has been in use.

A flexible rubber sleeve-type coupling connects the pump and motor, which gives great simplicity of construction, so that the pump can be stripped, cleaned and re-assembled in a matter of minutes, without the use of special tools.

The vacuum obtained is 0·1 mm. absolute pressure McLeod gauge. The pressure obtained is 10 lb./sq. in., with a swept volume of 28 litres/min. (1 cu. ft./min.).

Pulsometer Engineering Co., Ltd., Nine Elms Iron Works, Reading.

Stud Welding Attachment

An attachment for the Nelson stud welding tool has been developed by Crompton Parkinson, Ltd., which enables studs to be welded at right-angles to the tool. It therefore greatly facilitates the equipment's use in confined spaces, and is particularly useful when studs have to be welded to awkwardly located work faces, such as in rolled steel sections.

The attachment takes the place of the tool's standard chuck and leg assemblies, and is designed for easy fitting to the moulding of the tool by means of the three screws which are normally used to hold the quick adjustable leg bracket in position. The attachment can be fitted to the tool at any angle relative to the tool hand grip, thereby further facilitating the equipment's use for applications where operators have hitherto been unable to avoid obstructions. Apart from this, operation of the tool is exactly the same as with the standard arrangement. A patented fluxed stud is inserted in the chuck; a porcelain ferrule—the purpose of which is to prevent oxidation of the weld metal—is held round the end of the stud; and then with the now loaded

tool pressed sideways against the workpiece, the trigger button is depressed and the weld automatically completed. Studs of all diameters can be used in the special short chucks provided, and the standard leg and foot assembly can be employed at right-angles to the tool, shortened, if necessary, to the depth of the attachment.

Crompton-Parkinson, Ltd., Crompton House, Aldwych, London, W.C.2.

Oil and Water Separator for Air Lines

Bullows have recently produced a separator for which it is claimed that it completely removes oil from air lines at temperatures up to 300° F., and water from lines where condensation has taken place. Oil normally exists in compressed air in the form of very fine particles, and the result is an aerosol (a liquid in stable suspension in air) with which normal filters and separators are quite unable to deal.

The new separator consists of banks of long fibre felt pads, which have been specially produced for this duty. These pads become wetted by the passage of contaminated air, and cause the very fine particles of oil and water to coagulate, which increases the particle size, so that the oil and water are easily separated from the air stream. The filter is self cleaning and discharges collected oil and water through an automatic valve, so that it will operate for long periods without any attention, and without the filtering medium becoming saturated. It is available for wall mounting in two sizes, capable of dealing with 20 and 65 cu. ft./min., respectively, at 100 lb./sq. in.

Alfred Bullows & Sons, Ltd., Long Street, Walsall, Staffs.

Fume Scrubbing and Absorption Tower

Turner & Brown, Ltd., manufacturers of the well-known range of Turbro P.V.C. centrifugal fans, have now produced the Turbro fume scrubbing and absorption tower, claimed to be 100% efficient in the scrubbing of air saturated with sulphuric and hydrochloric acid, and 99·9% efficient with nitric acid. This unit has been specially designed for the elimination of fumes of these acids discharged in considerable quantities from various chemical and industrial processes.

The S/1 model, which is 8 ft. high × 2 ft. square, is capable of scrubbing fumes from 14–20 litres of evaporated acid per hour, and larger towers are in course of construction. The tower and internal components are constructed entirely from B.X. Cobex rigid vinyl acetate, and are therefore unaffected by the strongest acids.

The complete removal of the gases is carried out in three stages. The first is by absorption of the greater part of the gases in liquid, after which suspended globules of moisture are eliminated in a special eliminator section. Finally, any small traces of gases left after these two processes are completely neutralised by a chemical reagent contained in a tray. The chemical reagent tray is easily removed from the tower for the recharging which is necessary every 10–14 days, depending upon the amount of fume being scrubbed.

The Turbro tower can be supplied with a special fume cabinet, or alternatively as a single unit for incorporation into existing equipment.

Turner & Brown, Ltd., Bolton, Lancs.

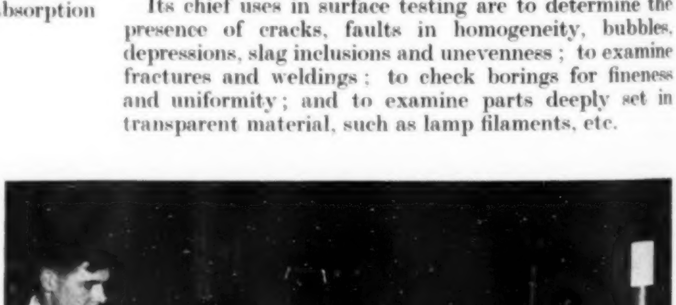
Stereoscopic Low Power Microscope

An industrial microscope with a number of unique features is one of the optical instruments now coming to the United Kingdom from the Carl Zeiss Works in Western Germany. It is known as the Technoscope, and is widely used in Germany, although there are at present very few in this country. Basically, the Technoscope consists of a stereoscopic microscope, to which a camera can be fitted, mounted on a portable "rollable" stand.

Its chief uses in surface testing are to determine the presence of cracks, faults in homogeneity, bubbles, depressions, slag inclusions and unevenness; to examine fractures and weldings; to check borings for fineness and uniformity; and to examine parts deeply set in transparent material, such as lamp filaments, etc.

Left—
Turbro fume scrubbing and absorption tower.

Right—
Technoscope with camera attachment in use.



Self-spraying pack for dye penetrant flaw detecting inks.

It is a complete unit with built-in light source (of variable intensity, with over-brilliance for photography) and filters. Five different magnifications (from $\times 6$ to $\times 40$) can be obtained simply by turning a knurled knob and, as the working distance remains the same, observation is not interrupted. The long working distance—some 8 in. between objective and object—permits examination down bores and channels, and the "rollable" stand, with multiple universal joints, enables the instrument to be used on completed structures in normally inaccessible positions. A further advantage is that anything seen through the binocular viewer can be photographed: stereoscopic pictures can be taken if required.

The industries in which the Technoscope is used include: iron and steel works and rolling mills; light metal works; boiler works; foundries; machine, turbine and motor factories; tool manufacture; the automobile industry; the metal goods industry; clock and watch manufacture; and the lamp and glass industry.

U.K. Agents: Degenhardt & Co., Ltd., 32 Maddox Street, London, W.1.

Stepless Controller

West Instrument, Ltd., now offer a new stepless temperature controller which does not incorporate any radio valves, for use with electric furnaces and other electrical loads. This instrument modulates the power input so that it equals the heat losses without the use of the contactors featured with conventional on-off systems. Thermal shock to heating elements is eliminated because power at a low level is supplied once the correct temperature is reached. The controller will vary power input to the load between 3% and 96% automatically according to demand, and units have been made from 300 VA. to 110 kVA.—single or three phase: high ratings are practicable. Multi-load units offer versatility, in that one unit can be used for several different loads, and on either 115 V. or 230 V. An adjustable band width, which can be adjusted at the front of the instrument to ½–4% of the scale range, enables a variety of unstable loads to be controlled. Manual reset allows correction of the "droop" or offset found in any heating system, and indicates the degree of offset.

West Instrument, Ltd., 1, Newman Street, London, W.1.

Crack in light alloy casting shown up by the use of Verimor detecting ink and Devmor developer.

Dye Penetrant Flaw Detection Inks in Pressurised Cans

Dye penetrant flaw detection inks are now being supplied by Manchester Oil Refinery (Sales), Ltd., in self-spraying pressurised cans. The dye penetrant method of non-destructive testing has been specifically developed for use where the absence of an electrical supply or suitably shaded area prevents the use of electro-magnetic or fluorescent techniques of testing. Spraying of the dye penetrant from a pressurised can has proved to be more economical and efficient than the conventional form of application by brushing.

By simply depressing a lever on top of the can, the dye is ejected in the form of a fine spray, which can be accurately directed onto the component under test. Two dye pentrants are available: Verimor, which is paraffin soluble; and Pentramor, which is water soluble. After spraying with the penetrant, the surplus is removed from the surface by a paraffin or water wash—depending on the penetrant used—before spraying with Devmor liquid developer. Flaws are revealed as brightly coloured lines on a smooth, matt-white background.

Manchester Oil Refinery (Sales), Ltd., Twining Road, Trafford Park, Manchester, 17.

Portable Vacuum Recovery Unit

Quasi-Arc, Ltd., have announced details of a portable vacuum recovery unit particularly designed for use with their Unionmelt welding process. In Unionmelt welding, the arc is submerged beneath a layer of granulated welding composition, and only a small percentage of this forms the protective slag covering to the weld. Unfused composition can be recovered for further use, and the vacuum recovery unit provides the ideal method for doing this. The operation of the machine is simple and quick, and its high efficiency ensures minimum waste. It can be supplied as part of a complete Unionmelt installation or as a separate unit, and incorporates features of special importance for the efficient handling of hot abrasive Unionmelt welding compositions. The machine is light and easily portable. The suction unit, powered by a 200/240 volt single-phase A.C. motor, is housed in the lid, so that the cylindrical body of the machine acts as the powder container.

Quasi-Arc, Ltd., Bilston, Staffs.

CURRENT LITERATURE

Book Notices

FOUNDRY VENTILATION AND DUST CONTROL

Proceedings of a British Cast Iron Research Association Conference at Harrogate, 1955. Foreword by Sir George Barnett, H.M. Chief Inspector of Factories. 257 pp., 264 illustrations, cloth. British Cast Iron Research Association, Bordesley Hall, Alvechurch, Birmingham. 60s. to non-members in the sterling area ; $10 in the dollar area.

THIS comprehensive report of the papers (and subsequent discussions) presented at the Harrogate Conference covers the entire field of dust control and ventilation in the foundry. It provides the fullest study so far available of the formation and dispersal of dust, and methods for its elimination or control in foundry operations. Some new experimental data have been incorporated since the holding of the conference.

The section on the formation of dust and fume in the ironfoundry gives the results of a fundamental enquiry based on systematic tests carried out by the Association over the last five years, into : (*a*) the formation and raising of dust and the behaviour of airborne particles ; (*b*) the influence on dust movement of the thermal currents originated by hot castings ; and (*c*) the effect of general air movement and cross draughts.

This material provides a basis of knowledge which, if correctly applied, can be used to solve problems of dust control arising during the normal sequence of foundry operations. An example of the practical interpretation of the results of this work is, in fact, provided by the detailed section on the control of dust at foundry knock-outs, where its principles are applied to formulate recommendations on the design of side-draught and down-draught knock-out ventilating systems. The method of determining the exhaust air volume required to give the appropriate air velocities to control thermal currents and cross draughts under specific operating conditions is fully explained, together with the effect of the hood profile and the height and size of the exhaust inlet. Typical examples are described and illustrated.

These methods of controlling dust at the knock-out are, of course, most suitable for foundries where some degree of mechanization exists, enabling castings to be brought to a common knock-out point and the sand to be mechanically reclaimed. A further paper on the use of core-assembly methods indicates another approach, particularly for the pit moulding foundry. By an economical change in the basic production method, the amount of dust can be reduced and the necessity for local handling is eliminated.

Many of the contributions deal with the control of dust during operations prior to, and during, grinding and fettling, and, in particular, these cover practical appliances for grinding and fettling, including stand grinders, swing-frame grinders, small portable grinders, pneumatic chisels, etc. The majority of these devices are now commercially available. Details are given of recommended designs of booth for the control of dust-bearing air streams during swing-frame grinding operations. The high pressure cleaning and de-coring of castings by both wet and dry methods are dealt with, and a simple wet spray bar for de-coring castings by hand is described.

Other papers cover general foundry ventilation, cleaning, maintenance and good housekeeping routines, the control of dangerous fume emissions, and methods of collecting cupola dust. There is also a paper detailing the choice of lighting methods now available to the foundry. Sir George Barnett, H.M. Chief Inspector of Factories has contributed the foreword to the volume and has also reviewed the problems of health and safety which arise in the foundry industry at the present day.

Every foundry concerned with meeting the ventilation clauses of the Iron and Steel Foundry Regulations (1953) which became effective on January 1st, 1956, or with the requirements of the Clean Air Bill, which will become law in 1956, will find much of interest and importance in this volume. The experimental work described is also of vital importance to the ventilating and consulting engineer, and others who are concerned with the utilization and commissioning of equipment for the suppression and control of dust in different industries.

THE ARC WELDING OF ALUMINIUM

Information Bulletin No. 19. The Aluminium Development Association, 33, Grosvenor Street, London, W.1. 92 pp., numerous tables and illustrations. 2s. 6d.

THIS is a Bulletin on practice, not intended directly to satisfy the needs of those concerned with metallurgical principles. The text begins by distinguishing between the older processes, requiring flux in order to remove the natural oxide film on aluminium, from the more recently developed inert-gas shielded-arc methods which obviate the use of flux.

Notes on factors governing the welding arc in general precede a brief description of the characteristics of aluminium in relation to arc welding ; and this leads, in turn, to a discussion of the weldability of aluminium alloys by the various arc processes. Tables, relating the cast and wrought alloys to the arc processes in order of suitability, list the appropriate filler rods and also show the mechanical properties to be obtained from the alloys unwelded and as-welded. References are made here to the use of welding to repair aluminium castings and to the increasing practice of assembling cast components or joining them to wrought components by welding.

The first principal section of the Bulletin, on inert-gas shielded-arc welding, deals first with the tungsten electrode and secondly with the consumable aluminium electrode process. As far as possible, information common to both subdivisions—for example joint preparation—is detailed once, and variations only are referred to in connection with the consumable electrode method. A series of tables giving recommended operating data for stated thicknesses of parent metal includes matters such as edge

preparation, pre-heating if necessary, electrode and filler wire diameter, current range, gas consumption and welding arc speeds. It is to be expected, of course, that results achieved from recommended practice will depend upon local conditions, such as whether the welding is done indoors or outdoors. The tabulated data are intended to assist operators in deciding, with minimum loss of time, the best practice for a particular piece of work.

The section on metal-arc welding—a thorough revision of the corresponding section of a former Bulletin—begins with recommendations on electrical equipment: choice of electrodes and fluxes are accompanied by tables giving recommended data for electrode diameter, current range and plate gap. Diagrams illustrate the type of joint recommended for metal arc welding and those not recommended unless the use of corrosive fluxes can be avoided.

The process of carbon arc welding, very largely superseded in this country, is briefly described. Atomic hydrogen welding, although involving the use of an electric arc, is regarded as belonging more appropriately to Information Bulletin No. 5 "The Gas Welding of Aluminium" and is therefore merely noted here as a cross-reference.

There are shorter statements on the process of working the weld, the inspection and testing of arc welds—including illustrations of radiographs and photo-macrographs—and on the arc cutting of aluminium. The book concludes with several sections of less operational interest but having considerable importance for welding supervisors and foremen—and also for designers—both in the selection of welding method and in the disposition of joints. These deal, for example, with welding economics, and with the strength and design of arc welded joints.

A SURVEY OF A CROSS-SECTION OF THE ELECTROPLATING INDUSTRY.

Published by The British Non-Ferrous Metals Research Association, Euston Street, London, W.1. Available free of charge from the Association. Applicants should state name of company and position held.

OVER the past two or three years The British Non-Ferrous Metals Research Association has been operating a special advisory service to electroplaters, designed to assist in increasing productivity and technical efficiency in the industry. The Association's investigators have visited the plating shops of a number of firms, by arrangement with the management, to make a detailed survey of the practice. Each survey has lasted from a few days to two or three weeks according to the size of the plant and, based on this survey, a confidential report was made available to the management of each firm indicating how productivity and efficiency could be increased.

Certain general conclusions and recommendations have come out of this work, and these have been embodied in a special report. This is not a technical report dealing with the actual plating processes employed, but is to some extent the converse of the now familiar Productivity Team reports issued by the Anglo-American Council on Productivity. It picks out the points where practice is at fault, where production is hindered, or where lay-out is bad, and indicates how improvements can be made. All concerned with the running of plating shops, whether managers, accountants, or shop supervisors, will find much of interest and value in a report drawn up by investigators familiar with, but sufficiently detached from, the industry to take a truly objective view of everyday practices which those engaged in the industry take for granted.

The Research Association was enabled to establish and operate this advisory service for electroplaters—which augmented and extended to non-members the ordinary technical consulting services available to member firms—by financial assistance received through the Conditional Aid Scheme for the use of Counterpart Funds derived from the United States economic aid administered originally by the Mutual Security Agency and latterly by the International Co-operation Administration.

Trade Publications

THE English Electric CMR 1600 range of heavy duty fabricated-frame D.C. mill motors designed for steel works auxiliary drives and cranes, hoists, etc., are featured in a 7-page illustrated leaflet. They comply with the American Association of Iron and Steel Engineers Standard No. 1, known as the "600" series, and are designed for 220/230 and 440/460 volts D.C., but for any given frame size correspond exactly in outputs and speeds to those for the American standards of 230 volts and 550 volts. Tables of performances and dimensions are included.

LEAFLET F.117, recently published by Evans Electroselenium, Ltd., 110, Potter Street, Harlow, Essex, describes the EEL Flame Photometer, which is a self-contained instrument for the accurate estimation of alkali metals in solutions. Full-scale deflection can be obtained with concentrates as low as 5 p.p.m. sodium, 10 p.p.m. potassium, 15 p.p.m. lithium and 50 p.p.m. calcium, but sensitivity control enables solutions of high concentration to be analysed.

ELECTRICAL Engineering Data Sheet 1300 : 338—"Button Contacts—Welding Technique," issued by Johnson, Matthey & Co., Ltd., describes the method of assembling these special composite contacts to their backings, using a conventional spot or projection welding machine. Compared with other methods of contact assembly, resistance welding with button contacts often has the advantages of economy, improved electrical conductivity, and better mechanical strength, with correspondingly greater resistance to stresses set up by changes of temperature. This publication is one of a series of data sheets on the company's products and services for the electrical engineering and allied industries.

WINN & COALES, LTD., Denso House, Chapel Road, London, S.E.27, have recently issued a publication devoted to the protection of structural steelwork against corrosion, using their Denso products. These are mainly tapes of various types which are applied to the cleaned metal surface in such a way as to make a complete air and waterproof seal. Where it is desirable to avoid the accumulation of fluff or dust or to alter the appearance the coating can be painted.

IN a leaflet dealing with continuous, normalizing, hardening and annealing furnaces, recently issued by

Gibbons Brothers, Ltd., Dudley, are featured illustrations of typical installations of disc hearth, walking beam and pusher types.

ALL who depend on photography for a livelihood will agree with the importance of the correct use of light filters. The ability of filters to modify the rendering of tone values and the contrast of originals is exploited in widely different ways. As an example, the photomicrographer uses them to increase the effective contrast or bring out the details of coloured specimens. With the title of "Light Filters," a recent publication of Kodak, Ltd., gives particulars of the Kodak and Wratten range for commercial, industrial, portrait, clinical, scientific, technical and general photography in black-and-white and in colour.

WE have received from Dowson and Mason, Ltd., Bulletin 1B on Car Bottom Furnaces, which gives a considerable amount of information of interest to engineers contemplating the installation of the larger types of heat-treatment furnaces. Illustrations and specifications cover gas, coal and oil-fired furnaces, with particulars of operating temperatures, maximum fuel capacities and the various methods that can be adopted for door operation. Details are also given of the various methods for moving bogies and the speed necessary for different purposes. Of particular interest is a list giving the dimensions of furnaces built recently which can be supplied at a lower cost than would operate if a different size were chosen. Other Bulletins recently published by the Company include P1, on steel storage tanks, which gives useful advice concerning the handling and storage of petrol and fuel oil; P2 on lubricating oil storage equipment; and P3 on diesel oil refuelling systems for the needs of modern transport. P5 is a reprint of a Bulletin published some time ago on liquid heating tanks, which find a variety of applications throughout the whole range of industry.

A LEAFLET recently issued by Hawksley S.M.D., Ltd., Slough, Bucks, illustrates the Hawksley S.M.D. system for constructing single-storey general purpose buildings whereby roof, walls and partitions are made up in modular units. This simplifies erection and provides an interior arrangement suitable for a variety of purposes. The leaflet also features abbreviated technical data concerning these buildings.

G.R. "GUNMAX" Basic Compositions are featured in a leaflet recently issued by General Refractories, Ltd., Genefax House, Sheffield, 10. These granular compositions possess high refractoriness, good packing density and the ability to adhere when applied to a hot lining. They are offered in three grades—C (chrome), MC (magchrome) and M (magnesite), and can be easily parged with a suitable paddle or shot by compressed air gun on to hot basic linings.

BULLETIN No. 158, issued by the Technical Service Department of Aero Research, Ltd., Duxford, Cambridge, is a reprint of an article on "The Place of Metal Bonding in Modern Aircraft Structures," by Ir. Edw. van Beek, Chief of Structures at the N.V. Koninklijke Nederlandse Vliegtuigenfabriek Fokker. This article was first published in *Technique et Science Aeronautiques*, journal of the Association Francaise des Ingenieurs et Techniciens de l'Aeronautique (A.F.I.T.A.), Vol. 4, 1955. It takes the form of a survey, from the purely structural point of view, of the possibilities and limitations of the metal bonding process, and of the best methods of making use of it, with maximum efficiency, in aircraft design.

A RECENT issue of *Evershed News* (Vol. 4, No. 2) contains an article on "Automatic Control of Soaking Pits," in which is described the installation of a plant of eleven producer gas-fired soaking pits at the Stocksbridge Works of Samuel Fox & Co., Ltd. Other articles deal with "Remote Indication and Control Equipment on Continuous Weighers"; "Modern Instruments for Plant Maintenance and Supervision" (with special reference to coal mines); and the "Earth Loop Resistance Tester."

"MAGNESIUM Alloy Pressure Diecastings" is the title of a publication recently issued by Stone-Fry Magnesium, Ltd. The endeavours of the company have led to the use of pressure diecastings for a wide range of products, from components of textile machines to cameras, and from ultra-light weight military equipment to moulds for mass-producing tiles. In this booklet details are given of properties, design, machining and finishing, which will be useful to those who are not already aware of the availability of diecastings in magnesium alloys.

THE Metals Division of Imperial Chemical Industries, Ltd., manufactures large tonnages of wrought nonferrous metals (copper, brass, aluminium and titanium) suitable for brazed or welded construction and, as a supplementary service, manufactures a wide range of brazing and uncoated welding rods. These rods are now marketed in suitable packages with the quality identified by a distinguishing label, and a new publication entitled "I.C.I. Welding Rods and Brazing Materials" has been produced to show the wide range of welding and brazing rods available.

THE second issue of the first volume of *Davy-United Engineering* includes an article by the Research Manager, R. B. Sims, entitled "A Note on the Calculation of Roll Force and Torque in Hot Mills," and articles on "The Measurement of Strip Tension" and "The Electronic Measurement of Load," by P. R. A. Briggs and W. H. Bailey.

Books Received

"Fluidization." Edited by D. F. Othmer. 231 pp., inc. index. New York and London, 1956. Reinhold Publishing Corporation and Chapman & Hall, Ltd. 56s. net.

"High-Temperature Technology." Edited by I. E. Campbell. 526 pp., inc. author and subject indexes. New York and London, 1956. John Wiley & Sons, Inc., and Chapman & Hall, Ltd. 120s. net.

"Temperature: Its Measurement and Control in Science and Industry." Vol. II. Edited by H. C. Wolfe for the American Institute of Physics. Papers presented at the Third Symposium on Temperature, in Washington, D.C., October, 1954. 467 pp., inc. author and subject indexes. New York and London, 1955. Reinhold Publishing Corporation and Chapman & Hall, Ltd. 96s. net.

"Iron and Steel Today." By J. Dearden. 2nd Edition. (The Pageant of Progress Series, No. 13). 271 pp., inc. index. London, 1956. Oxford University Press: Cumberlege. 12s. 6d. net (in U.K. only).

LABORATORY METHODS
MECHANICAL · CHEMICAL · PHYSICAL · METALLOGRAPHIC
INSTRUMENTS AND MATERIALS

JULY, 1956 Vol. LIV, No. 321

A Magneto-Electric Method for Detecting Defects in Cast Aluminium Rotor Windings for Squirrel-Cage Motors

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Research Department, Metropolitan-Vickers Electrical Co., Ltd.

The article describes a method developed by the authors for detecting hidden defects in the cast aluminium rotor windings of squirrel cage induction motors. The method involves turning the rotor slowly at a steady speed in a steady magnetic field, and noting any variations in the cross-field due to the induced current: variations indicate defects in the casting.

In the manufacture by the M.V.E. Co., Ltd., of squirrel-cage rotors with cast aluminium windings, molten aluminium is fed into the closed slots of a stack of punchings forming the rotor core. In this process, the punching stack is held on a mandrel enclosed in a mould which is rotated during the casting operation: on cooling, the aluminium forms a homogenous assembly of solid bars in the slots, and short circuiting rings at each end of the stack of punchings or core (see Fig. 1). This procedure results in a most satisfactory squirrel cage rotor which is robust, has good appearance, provides adequate support of punching teeth at the periphery, has less windage, and is quieter than the type having an assembly of copper bars and rings joined together by conventional methods. In addition, it may be produced more quickly, it permits greater variety and choice of design characteristics, and it is free of the troubles associated with the joining of copper bars to copper end rings.

Fig. 1. Rotor F in the as-cast condition, prior to machining.

Since, however, much of the aluminium casting is hidden within the punching slots, visual checks on casting quality can only have a limited value. Hence, it was necessary to devise some simple method for checking the quality of castings before proceeding with motor assembly, thus providing an early routine check on manufacture. Discrimination between good and bad rotors has hitherto been made in the final testing procedure of the finished motor, incurring unnecessary expense where castings were found to be defective. Fig. 2 shows a rotor which has melted away its windings while running, due to casting defects. This article describes a practical answer to the problem which is now being incorporated in the production process.

Method Adopted

As a result of earlier work by M-V. Research Department, a method* devised for testing aluminium billets for defects was available, with slight modification, for application to rotor windings. The modified test method may briefly be described as follows (see Fig. 3) :—

The motor rotor to be tested is placed on end on a horizontal, non-magnetic turntable, so that its axis is substantially vertical. It is carefully centred and rotated about its axis at approximately one revolution per second. The rotor is located centrally between the poles of a strong, permanent magnet, or a constant electromagnet, *NS*, so that a steady magnetic field of a few hundred gauss passes across the air gaps and through the diameter of the rotor core at approximately the middle zone of the stack of punchings. The magnet poles are preferably elongated at their faces, so as to scan several inches of the rotor bars under the pole faces.

A detector coil is fixed with its axis at right angles to that of the magnet field, so as to scan the rotor surface midway between the pole faces. This coil is connected through a suitable sensitivity control to a moving coil pointer instrument.

As the rotor is revolved at a constant speed within the steady magnetic field, an E.M.F. of a few millivolts is generated up each rotor bar, in turn, and down the diametrically opposite bar passing the poles. This gives

* British Patent No. 573,152.

Fig. 2.—Defective rotor A, which failed in service.

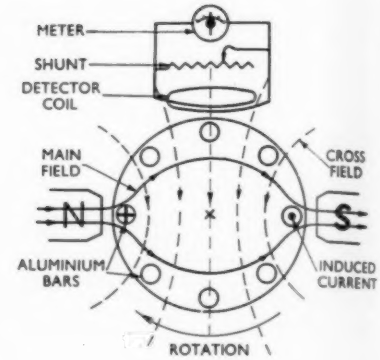

Fig. 3. — Diagram illustrating the principle ofthe magneto-electric method of test for cast aluminium squirrel-cage rotors.

rise to a current of about 50 amp., which flows up each bar in turn, along the two halves of the top end ring in parallel, down the diametrically opposite bar, and back through the two halves of the lower end ring in parallel. Thus a closed-current loop is formed, giving rise to an " armature reaction " cross-field which links the detector coil. Any change in the value of the induced current is reflected in the detector coil flux linkage, and will be shown on the meter.

If the bars and end-rings have constant resistance, a substantially steady cross-field is maintained linking the detector coil, and hence no meter signals appear. This is taken as evidence of sound casting in the rotor. If defects are present of sufficient size to affect the conductance, the induced electric current, and hence the value of the magnetic cross-field, fluctuates, giving detector instrument indications having a varying pattern repeated for each half revolution. As these are readily recognised, good and bad rotor castings are quickly sorted, provided all the slots in a given rotor are of the same shape and size and the speed of revolution is constant.

Interesting secondary effects result from this test procedure. With a sound rotor revolving, but not accurately centred on the turntable, the air gap between the detector coil and rotor surface varies. Then the fluctuating field flux produces cyclic detector signals varying once per revolution. Hence accurate centering should be achieved to avoid such confusing signals. However, it has been found that the use of an exactly similar detector coil on the opposite side of the rotor, if connected in series with the first detector coil, largely eliminates eccentricity effects.

A correctly centred faulty rotor, producing fluctuating cross-fields, is found to modulate the main flux in the air gap. Hence, a search coil placed therein forms an alternative means of actuating the detector instrument. Similarly, the fluctuations in the end ring currents can be used to indicate faults.

Results on Faulty Test Rotors

Eight faulty rotors were selected for trial purposes, each one being suspect for one reason or another. They were marked *A*, *B*, *C*, *D*, *E*, *F*, *G* and *H* for identification purposes. This selection was the best available at the time, ranging from very bad to apparently good cast windings.

Induction Tests (see Fig. 3)

Each rotor was centred upon a 12 in. dia. brass turntable and revolved at one revolution per second through approximately 500 gauss air gap flux density through pole shoes $4\frac{1}{4} \times \frac{3}{4}$ in., provided by two Alcomax permanent magnet blocks. A detector coil of 1,500 turns about a 4 in. diameter was placed approximately 1 in. from the surface of the rotors, and mid way between the poles. The search coil was connected to a quick-acting moving coil millivoltmeter suitably shunted to control its sensitivity. The comparative records of the readings of the millivoltmeter are given in Fig. 4 for reasonably good and very bad rotors, i.e., rotors *E* and *D*, respectively.

Other Test Methods

(1) A bar-to-bar D.C. conductance test was made at 500 amp. on rotors *D* and *E*, the potential across each bar being measured by a moving coil millivoltmeter. The relative conductance of each bar was calculated by

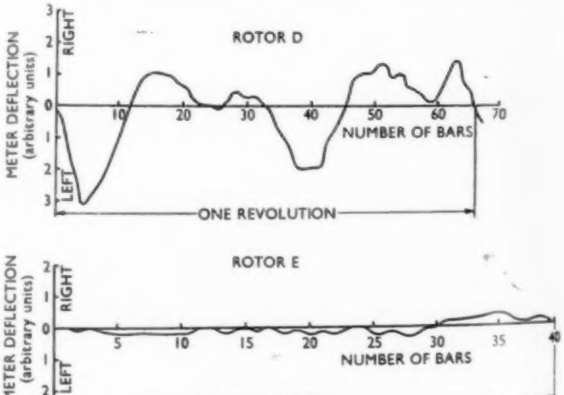

Fig. 4.—Comparison of records obtained on a " bad " rotor (D) and a " good " rotor (E).

Fig. 5.—Partially turned rotor D, showing bar defects.

Fig. 6.—Close-up of bar defects in rotor D. The dark zones are the iron punchings and the light zones the cast aluminium bars.

dividing the total current by the millivoltmeter deflection. Rotor *E* showed little variation from bar to bar, but rotor *D* showed large variations. This is confirmatory of the induction test results.

(2) A similar test was made on rotor *D*, but the current in each conductor was measured by a magnetometer reading of the flux density across the mouth of the slots in turn. Again, large variations were found from slot to slot, some slot bars having no current flowing in them, which indicated complete fracture.

(3) A rough ultrasonic transmission test from one end-ring to the other was made over every fifth bar of rotor *D*. Large variations in transmitted energy were noted. Good bars would be expected to transmit readily, defective bars would transmit poorly or not at all. Many zones had zero transmission, it was found, suggesting completely severed bars. The zones suspected as faulty from the above tests, which agreed very well amongst themselves, were marked, and the rotor *D* turned in a lathe on its cylindrical surface with $\frac{1}{32}$ in. cuts until faulty bars were revealed. These faults appeared as contraction cracks across the conductor section over almost half the conductors, grouped about one side of the rotor (see Figs. 5 and 6).

The results obtained by all the methods described above were carefully compared ; they were found to give mutually confirmatory conclusions.

Summary of Results

All the rotors in this test series had some kind of defect. These have been summarised in Table I.

TABLE I

Rotor Identification	Nature of Defects Found	Illustration
A	Not tested. Overheated in service at defects. Aluminium melted	Fig. 2
B	Bars damaged	—
C	Some empty slot necks	—
D	Many contraction cracked bars	Figs. 4, 5 and 6
E	Some empty slot necks	Fig. 4
F	Cracks in end-ring	Fig. 1
G	Almost sound rotor	—
H	Defective end-ring, cracked bars	Figs. 7 and 8

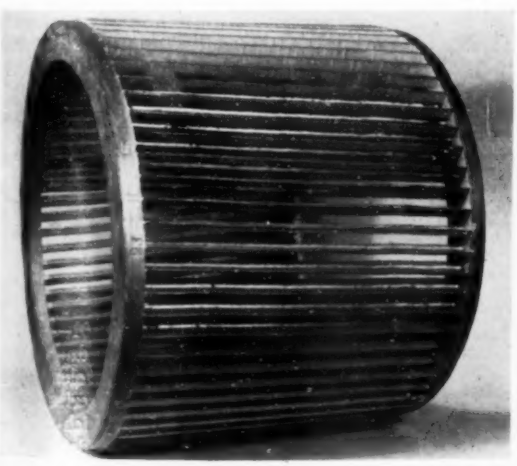

Fig. 7.—Squirrel cage freed from iron core by dissolving the latter in acid. Rotor H.

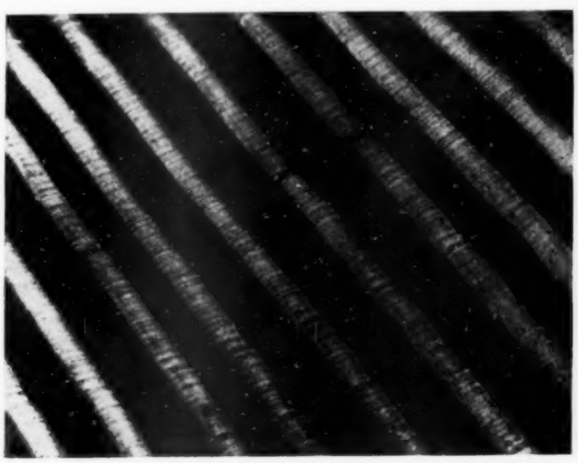

Fig. 8.—Close-up of cracked bars inside rotor H after dissolving the iron core.

Rotor H was classed as fairly bad when tested, the results falling somewhere between those of rotor E and rotor D (see Fig. 4). It was seen to have a radial crack in one end-ring, and its squirrel cage was freed from the iron core by dissolving the latter in acid (see Fig. 7). This revealed several badly cracked bars on the inside section, as shown in Fig. 8.

Conclusions

The induction test method described appears very suitable for shop testing of cast aluminium squirrel cage rotors. It is essentially a test for uniformity of conductance, utilising an internally generated electric current of a very low frequency. Hence there is no electrical supply problem, load conditions are simulated, and the display instrument is simply and quickly interpreted. The cost of the equipment is low and needs little maintenance: it can be operated by unskilled workers.

The method will indicate roughly the particular portions of the windings which are defective, although this is not normally necessary, as any indication of non-uniformity warrants rejection of the rotor. It will not indicate slot necks devoid of metal at the rotor surface, unless these seriously reduce the effective conductance of the conductors. In any case, these defects can be detected by visual inspection.

The test would fail in the unlikely event of every slot bar being defective to an extent which rendered its conductance exactly equal to the others. In this case, the detecting instrument would remain steady, but subsequent test-bed figures would reveal such a defect.

Acknowledgments

The authors express thanks to Dr. Willis Jackson, F.R.S., Director of Research and Education, and to Mr. B. G. Churcher, M.Sc., M.I.E.E., Manager of Research Department, Metropolitan-Vickers Electrical Co., Ltd., for permission to publish this paper.

Society of Instrument Technology A.G.M.

At the Annual General Meeting of the Society of Instrument Technology, Ltd., held at Manson House, London, W.1, on the 28th May, 1956, the President, Mr. A. J. Young, of I.C.I., Ltd., said that the Society continued to flourish, as indicated by the rise in membership of 225, the highest yearly increase to date. It was even more satisfying to report the increased activities during the past year; notably the Joint Conference with the Institute of Chemical Engineers to stimulate interest in instrumentation and automatic control in the chemical industry; the discussion meetings arranged in conjunction with the Instrument Industries Exhibition at Earls Court, where education had been the main theme; and the recent most successful conference at Cambridge on "Plant and Process Dynamic Control," where fundamental research had been the main topic.

Continuing, Mr. Young paid tribute to Dr. H. Moore, C.B.E., who, though remaining Hon. Treasurer for the time being, had resigned the Editorship of the Transactions. Dr. Moore had been elected an Honorary Life Member of the Society, and it was hoped that his valuable experience and advice would be available to Council and Officers in the future. The new Editor was Mr. N. Ream, of the National Physical Laboratory.

With the increase in activities and prosperity, the Society had been fortunate enough to obtain office accommodation in S.I.M.A. House. Owing to his other commitments, however, Mr. R. D. Smyth had been unable to continue as full-time Secretary and had been succeeded by Commander A. A. W. Pollard. Mr. Smyth was thanked for his invaluable work.

Dr. H. Moore, Hon. Treasurer, then presented the Accounts for the year 1955. Revenue from members' subscriptions was up by £265, and for the first time the Society had money invested earning interest. Despite the ever increasing cost for publishing the *Transactions*, the excess of income over expenditure was about £900, a very satisfactory figure. However, part of this sum was made up of non-recurring items and, with the opening of the London office, the Society had taken on formidable commitments. It was all the more necessary, therefore, to obtain more revenue by increased sales of the *Transactions*, more Patrons (of which there are now 26), and more members.

The following were elected to Council to replace retiring members:—User Group: Mr. G. C. Eltenton (Shell Refining & Marketing Co., Ltd.) and Mr. R. Riley (Costain—John Brown, Ltd.) *vice* Mr. P. F. Cook and Dr. R. Jackson; Manufacturers: Mr. R. S. Medlock (Director, George Kent, Ltd.) *vice* Mr. H. R. Walton. Education & Research: Professor A. Porter (Imperial College of Science) *vice* Dr. G. D. S. MacLellan.

New British Chemical Standards

Bureau of Analysed Samples, Ltd., Middlesbrough, announce a new list of standard samples which includes the following new items:—

- B.C.S. No. 266. Permanent magnet alloy (13% Ni, 24% Co, 1% Nb)—for complete analysis.
- B.C.S. No. 174/1. Basic slag—for complete analysis, including citric soluble P_2O_5.
- B.C.S. Nos. 248 and 249. Basic slags—for sulphur only. These samples are intended as standards for the determination of sulphur by combustion.
- B.C.S. No. 268. 5% silicon-aluminium alloy (near to B.S.1490 : LM16)—for complete analysis.
- B.C.S. No. 269. 34% alumina firebrick—for complete analysis.
- S.S. Nos. 11–17. A unique series of seven spectrographic standards of mild steels in the form of ¾ in. diameter rods containing increments of a large number of residual elements. Chemical standardization at present extends to the following: Ni, 0·01–0·23%; Cr, 0·02–0·18%; Mo, 0·01–0·19%; Cu, 0·01–0·21%; W, 0·01–0·28%; Co, 0·01–0·25%; Sn, 0·01–0·11%; and V, 0·01–0·10%. It is hoped, in due course, to standardise the following additional elements—Ti, Al, Zr, Nb, Ta, Pb and B. This series is also available in the form of turnings which may be used as chemical or photometric standards.

Copies of the new list, No. 386, giving details of all the standards available, may be obtained post free from the office of the Bureau, Newham Hall, Middlesbrough.

Liquid-Liquid Extraction Procedures in Inorganic Analysis

A Review of Practical Applications with Particular Reference to Metallurgical Analysis

By T. S. West, B.Sc., Ph.D., A.R.I.C.

Department of Chemistry, University of Birmingham

The growing complexity of modern materials has led to difficulties in their chemical analysis resulting from the increasing multiplicity of other substances accompanying the constituent to be determined. The development of new separation methods has made possible the continued use of classical analytical procedures in the face of increasing competition from physical methods, and in the present series of articles the author surveys liquid-liquid solvent extraction techniques in inorganic analysis, with particular reference to metallurgical analysis. In the sixth of the series, the author discusses the extraction of Group VIa, VIIa and the transition metals.

(*Continued from page* 294 *of the June issue of* METALLURGIA)

EXTRACTION OF GROUP VIa METALS (Cr, Mo, W, U)

Chromium

CHROMIUM is not extracted from hydrochloric acid medium by diethyl ether under the conditions which favour the complete removal of iron, molybdenum, etc. No reports of extraction from bromide or iodide solutions could be found, and Kitahara[37] has reported that no extraction occurs from fluoride solution. Aven and Freiser[43] comment that chromium may be extracted as its thiocyanate by tri-*n*-butyl phosphate. Appreciable amounts of Cr^{VI} are extracted from 8N nitric acid by diethyl ether, but the system is unstable, and it does not appear to have been used as a means of separating chromium from other ions in aqueous solution. Chromium is frequently separated from vanadium by extracting the latter with chloroform and 8-hydroxyquinoline, but more recently another method has been proposed in which the chromium is preferentially extracted by methyl isobutyl ketone from a hydrochloric acid medium. With both metals in their highest oxidation state, separation factors of *ca* 4,000 were obtained using low acid concentrations in the temperature range 0–25° C. Weinhardt and Hixson,[102] who were concerned with industrial application of the method for the treatment of chromite and chrome bearing ores, suggest that the favourable factor may be useful in developing an analytical method. The dichromate is easily stripped from the organic phase by equilibrating several times with distilled water.

Another recent method involving the extraction of a simple inorganic compound of chromium uses ethyl acetate as the solvent. This method also gives a good separation from vanadium. The formation of blue perchromic acid by the action of hydrogen peroxide on dichromate was reported in 1847.[103] In 1946, Foster[104] showed that the perchromic acid could be extracted from vanadium products by ethyl acetate. Foster's method was subsequently examined by Brookshier and Freund.[105] These workers found that a single extraction gave 80% recovery of chromium at *p*H 1 and 95% at *p*H 2. At *p*H 4, however, only 30% recovery was obtained. Amounts of chromium from 0·4–4 mg. (a concentration range of 0·000154–0·00154 molar) were extracted. Maximum recovery of chromium was obtained when the hydrogen peroxide concentration was 0·02M. The percentage recovery decreased slowly above this concentration. Virtually complete recovery of chromium occurred in a series of three extractions.

The stability of perchromic acid alters markedly with temperature. Maximum stability (and therefore maximum extraction) occurs below 10° C. and the perchromic acid is considerably more stable in the ester phase than in aqueous solution. The authors claim that iron, mercury, vanadium, titanium, nickel, molybdenum, etc., do not interfere to any marked extent.

$Chromium^{III}$ is readily extracted from aqueous solution by a reagent consisting of cupferron in ethyl acetate. Chromium does not react with dithizone and although a coloured compound is formed with diphenylcarbazide, it does not appear to be extracted by the more common solvents.[106] The complex formed with sodium diethyldithiocarbamate extracts well into chloroform at *p*H values up to *p*H 6·3, but not at higher values.[101]

Molybdenum

Molybdenum is extracted from 6N hydrochloric acid to the extent of 80–90% by diethyl ether. Kitahara[36] reported *ca* 6% extraction from 6·9N hydriodic acid using diethyl ether, and Irving and Rossotti[38] record that less than 1% is extracted from 1·5N acid containing an excess of potassium iodide. Kitahara obtained 9·7% extraction from 30% hydrofluoric acid solution. In the presence of reducing agents such as stannous chloride, $molybdenum^{V}$ can be extracted as the thiocyanate. The intense red colour of this compound has been used considerably in absorptiometry, since not only does the extraction procedure increase the sensitivity of the reaction, but it also provides an excellent separation from many other metals. Many solvents such as diethyl ether, butyl acetate, and cyclohexanol have been used. Tungsten,

which extracts under similar conditions as a purple complex, can be retained in the aqueous layer by adding tartrate or citrate, without decreasing the extractability of the molybdenum. Platinum is reduced by stannous chloride to chloroplatinous acid, and this accompanies the molybdenum into the ether layer. The most favourable acidity for the operation appears to be in 5% acid. Iron, aluminium, titanium, manganese, nickel, cobalt, uranium, and tantalum are said not to be extracted. A small amount of chromium is extracted when it is present in preponderant quantities. The same considerations apply to vanadium. Reasonable amounts of phosphate and fluoride in no way interfere, but large amounts of the latter are known to depress formation of the molybdenum thiocyanate compound.

Potassium ethyl xanthate, $C_2H_5O.CSSK$, imparts a red colour to solutions of molybdenum. Feigl has assigned the formula $MoO_3.[(CS.(SK)\ (OC_2H_5)]_2$ to the compound. Malowan[107] showed that molybdenum could be extracted in this form from faintly acid solutions: several heavy metals interfere. Sodium thiosulphate also forms an extractable lilac-coloured reduction product with molybdenum in acid solution.[108] Ether and ethyl acetate may be used as solvents for this extraction.

Gentry and Sherrington[16] obtained quantitative extraction of 200μg amounts of molybdenum with a 1% solution of oxine in chloroform over the range *p*H 1·6–5·6. Tartrate prevented complete removal of molybdenum when present in the aqueous solution. Sandell[29] observes that the corresponding tungsten compound is insoluble in chloroform.

Molybdenum^{VI} forms two precipitates with cupferron. Cup. MoO_3 and $(Cup)_2MoO_3$. The first precipitate is insoluble in benzene, but the second is readily soluble in benzene and chloroform. Furman, Mason and Pekola[17] state that, at *p*H>1, amounts of molybdenum of the order of 5 g./litre may be extracted quantitatively by chloroform and cupferron, and that quantitative extraction also occurs from 18% hydrochloric acid when ethyl acetate containing cupferron is used as reagent.

The green compound formed between 3, 4-toluene dithiol in hydrochloric acid can be extracted by amyl acetate.[109] In the presence of concentrated hydrochloric acid [sp. gr. 1·075] and a small amount of phosphoric acid at room temperature, the interference of tungsten is eliminated[110] and a 1% solution of the dithiol in amyl acetate may be used for the extraction. The method is the basis of an excellent colorimetric method. No interference is found from cobalt, vanadium, tin, lead, nickel, manganese, aluminium, etc. Small amounts of iron are extracted, but can be accounted for in the absorptiometric method by a steady blank in analysing steels for molybdenum. Copper, when present, causes the amyl acetate layer to become black and cloudy, but it can be stripped from the organic phase simply by washing with strong hydrochloric acid.

Tungsten

Whilst appreciable amounts of molybdenum are extracted from hydrochloric acid by diethyl ether, particularly in the presence of large amounts of ferric chloride, tungsten is not extracted to any appreciable extent. Irving and Rossotti[38] reported that less than 1% of tungsten^{VI} was extracted from 1·5N hydriodic acid by diethyl ether. Like molybdenum, tungsten may be extracted from aqueous solution in the presence of stannous chloride and ammonium thiocyanate. The operation does not provide as good a method of isolation as in the case of molybdenum, however.

Tungsten does not react with dithizone, and according to Sandell[29] the oxine precipitate is insoluble in chloroform. According to Furman *et al.*[17], the extraction of tungsten from 10% hydrochloric acid by cupferron in ethyl acetate is only partly complete.

Uranium

The extraction of uranium from aqueous solution is a method used extensively for the purification of uranium for use in atomic energy projects. However, this extraction is a commercial large-scale process, and although it illustrates the potential value of extraction methods in general, it does not really concern us here.

Uranium is not extracted by diethyl ether from hydrochloric acid solution or from solution in the other halogen acids, but a small amount (*ca* 5%) of uranium^{VI} can be extracted from a 50% solution of ammonium thiocyanate in 0·5N hydrochloric acid.[42]

Because of its importance in atomic energy projects, the ether extraction of uranyl nitrate from nitric acid solution has received widespread attention from all aspects of the subject. The extraction was first advocated in 1842.[111] Many solvents other than ether have been used—esters, ketones, alcohols, and other ethers, i.e., those solvents containing donor oxygen atoms—but solvents such as benzene and carbon tetrachloride are incapable of extracting the uranyl nitrate. The presence of salting out agents such as calcium nitrate, ferric nitrate, and ammonium nitrate has been found to increase the extractability to a marked degree. Cerium^{IV} and thorium extract appreciably under the conditions favouring extraction of uranium. The interference of cerium can be readily overcome by reduction to cerium^{III} through addition of sodium nitrite to the solution. Anions such as chloride should be absent, since not only do they decrease the tendency for uranyl nitrate to extract, but they also increase contamination from ferric iron, etc. Large amounts of sulphate interfere, and phosphate also reduces the extractability of the uranium. The addition of calcium nitrate prevents interference from the former anion, whilst ferric nitrate or aluminium nitrate may be added to prevent the interference of the latter.

Uranium may not be extracted by dithizone and chloroform, but the precipitate formed by oxine with uranyl salts is said to be soluble in chloroform,[29] and may presumably be extracted. Sodium diethyldithiocarbamate may be used in conjunction with chlorofrom to extract uranium from solution, but not at low *p*H values.[106] Furman[17] *et al.* note that while uranium^{VI} may be extracted in milligram amounts from very concentrated aqueous solutions in 10% hydrochloric acid by cupferron in chloroform, the compound formed with uranium^{IV} is soluble in chloroform, but is incompletely extracted from aqueous solution. Macro amounts are extracted from sulphuric acid solution by cupferron in ether.

EXTRACTION OF GROUP VIIa METALS (Mn, Tc, Re)

Manganese

Manganese is not extracted from aqueous hydrochloric acid solution by diethyl ether, and only negligibly small amounts are removed from a nitric acid medium. As far

as is known, manganese is not extracted by diethyl ether from solutions containing thiocyanate, hydrobromic acid, hydrofluoric acid, or hydriodic acid. Indeed the only method proposed for the extraction of manganese from aqueous solution as a purely inorganic compound appears to be that of Hornig, Zimmerman and Libby.[112] These authors showed that permanganate is practically quantitatively extracted by pyridine from an aqueous solution 4M in potassium hydroxide. The distribution coefficient is of the order of 8,000. Willard and Perkins[73] found that permanganate could be extracted from aqueous solution by tetraphenyl phosphonium chloride and chloroform, but no details were given of the actual extraction procedure.

Gentry and Sherrington[16] state that, in the presence of tartrate, manganese^{II} is extracted from aqueous solution by a 1% solution of oxine in chloroform over the *p*H range 7·2–12·5. Dithizone may be used in conjunction with chloroform[29] to extract manganese^{II} from aqueous solution at *p*H 11, but the extract is very unstable. According to Furman[17] and his co-workers, the precipitate formed by cupferron with divalent manganese in aqueous solution is soluble in ether. The method may, perhaps, be used as an extraction procedure. West, Lyons and Carlton[9] have found that manganese is extracted from aqueous solution *p*H 6·3–10·3 by a solution of capric acid or butryic acid (5%) in benzene. In the latter case, only copper and iron are extracted along with the manganese.

Rhenium and Technetium

Rhenium and technetium may be extracted from alkaline solution by pyridine according to Goishi and Libby.[113] They must be present as pertechnetate or perrhenate ions. Technetium is more readily extracted from 4N sodium hydroxide solutions than is rhenium. The distribution coefficient for the former is 778, while for the latter it may vary from 30 to 255, according to the amount of the ion in solution. Both ions may be extracted by chloroform and phenyl phosphonium chloride.[73] Tribalat[114] has shown that rhenium may be separated from molybdenum by extraction of the tetraphenyl arsonium salt with chloroform. The extraction is said to take place readily from a bicarbonate buffered medium (*p*H 8–9). The extraction is proportional to the concentration of the tetraphenyl arsonium chloride in the aqueous layer, and inversely proportional to the concentration of free chloride ions. The latter ions increase the tendency for the reagent to extract. The rhenium may be back extracted from the chloroform phase by shaking with strong hydrochloric acid solution. Hurd and Hiskey[115] reported that molybdenum could be extracted along with rhenium from thiocyanate solution by ethyl ether. The rhenium is probably extracted as ReO $(CNS)_4$. Tribalat and Beydon[116] have more recently published details of the extraction of technetium by chloroform and tetraphenyl arsonium chloride from alkaline solution.

EXTRACTION OF THE TRANSITIGN METALS (Fe, Co, Ni)

Iron

The extraction of ferric chloride from hydrochloric acid solution by means of various ethers and amyl acetate, and the extraction of the ferric acetylacetone complex, have been dealt with previously by the author in this journal[27]: there will, therefore, be no further mention of these particular methods in the following paragraphs. Ferric iron is not extracted from nitric acid solution, and only 60% is extracted from 6N hydrobromic acid. Aven and Freiser[43] have published a method for the extraction of ferric iron as the thiocyanate complex, using tri-*n*-butyl phosphate as solvent. Their method, based on earlier work by Clifford, Jack, and Lewis, enables iron to be separated from antimony, gold, molybdenum, thallium, tin, etc., which metals usually accompany iron into an ether phase from aqueous hydrochloric acid solution, but cobalt, copper, chromium, and zinc are extracted from the thiocyanate solution by the butyl phosphate. It would appear from these results, therefore, that two extractions, one with amyl acetate from hydrochloric acid solution, and one with tributyl phosphate from thiocyanate solution, should furnish a virtually specific method for the isolation of iron. Aven and Freiser obtained 97·4% removal of ferric iron from aqueous solution in one operation when the thiocyanate : iron ratio was not less than 6 : 1. The amount of iron extracted was found to decrease with increase of temperature. The extraction also decreased with rise in acidity, the optimum *p*H value being 1. The extraction was further studied by Melnick, Freiser and Beeghly.[117] The transfer of the iron to the organic phase takes place very rapidly (30 seconds being sufficient), with little rise in temperature. No discernable volume changes occur, and the distribution coefficient is independent of the iron concentration over the range 0·04M–0·4M. Butyl phosphate is less dense than the aqueous phase, but Freiser and his co-workers found that a 50% solution of the ester in carbon tetrachloride was almost as efficient as the ester alone in extracting the iron, whilst it is more dense and therefore more easily used in an ordinary extraction funnel for repeated extractions of the aqueous solution. Musakin[118] used amyl alcohol as solvent to extract small amounts of ferric iron from a slightly acid 10% potassium thiocyanate solution. Other authors have advocated the use of ethyl acetate,[119] amyl alcohol-ether,[120] ether-monobutyl ether of ethylene glycol,[121] etc., for solvent extracting the thiocyanate complex, but these are mainly concerned with the development of a colorimetric method of determining ferric iron.

In their review of the extraction of cupferrates, Furman, Mason and Pekola[17] state that ferric iron is extracted quantitatively from 10% hydrochloric acid by cupferron and chloroform, or by ethyl acetate or ether, leaving not even a spectrographic trace in the aqueous phase. Extraction also takes place favourably from 10% sulphuric acid by cupferron and diethyl ether. Many authors have reported use of the cupferron extraction as a means of removing undesirable amounts of iron from solution. One such paper is that of Strafford and Wyatt.[122] In evolving an aurintricarboxylic acid method for aluminium, interference due to ferric iron was overcome by extracting the latter as its cupferrate into chloroform from 4–5N sulphuric acid. The excess cupferron was extracted sufficiently well to prevent interference with the aluminon method, whilst no extraction of aluminium occurred when the acidity was in excess of 4N. In developing a method for the microanalysis of silicate rocks, Miller and Chalmers[45] found the efficiency of the common solvents was barely adequate for extracting ferric and titanium^{IV} cupferrates, and they proposed *o*-dichlorobenzene as a much superior solvent. With this solvent, it was found that only 85% of ferrous iron could

be extracted, and since reduction always occurred when fusing with bisulphate in platinum ware, it was necessary to re-oxidise the iron in solution. Titanium, zirconium, and vanadium were removed along with the ferric iron. The theory of the cupferrate extraction has been developed by Furman *et al*[17] and by Sandell and Cummings.[123]

Using a 0·01M solution of oxine in chloroform, Moeller[15] obtained complete extraction of ferric iron from aqueous solution in the pH range 1·9–3·0, whilst Gentry and Sherrington[16] used a 1% solution of the reagent in chloroform to obtain extraction in the range 2·5–12·5 in the presence of tartrate. Ferric iron does not form a complex with dithizone, although it oxidises it, but ferrous iron forms an intense violet red complex, which can be extracted into carbon tetrachloride over the range pH 6–7.

Lacoste, Earing and Wiberley[101] showed that both ferrous and ferric ions could be extracted from aqueous solution by sodium diethyl dithiocarbamate and chloroform over a wide pH range (*ca.* pH 0–10). Bismuth, cobalt, nickel, chromium, and uranium are also extracted.

The *o*-phenanthroline method for the absorptiometric determination of ferrous iron is a well-established procedure. It is, however, limited by the fact that cations other than iron react to form coloured products. In determining trace amounts of iron in vanadium, chromium, and nickel, Margerum and Banks[124] overcame the usual interferences by extracting the ferrous complex as the perchlorate salt into nitrobenzene. Several solvents, such as chloroform, etc., will extract the water-insoluble ferroin perchlorate, but several extractions are necessary, and, on standing, the perchlorate precipitates. Nitrobenzene is a much superior solvent, and the high distribution coefficient permits quantitative removal of the ferrous iron from the aqueous phase in one operation. The slow reaction rate of chromium, vanadium, and nickel with *o*-phenanthroline permits quantitative removal of the iron before these can interfere.

More recently, however, Powell and Taylor[125] have shown that it is not necessary to rely on the presence of perchloric acid to ensure extraction of the ferroin complex, for they have shown that the presence of a long chain alkyl sulphate or sulphonate will promote the extraction of both the ferrous *o*-phenanthroline and ferrous α, α'-dipyridyl complexes from aqueous solution into chloroform. Powell and Taylor ascribe the extractability of the complexes, in the presence of those "promoters," to the neutralisation of the electrostatic charge on the cation by the organic anion, and the presence in the latter of a large non-polar group. It was found that, whereas an excess of the promoter was necessary to ensure complete extraction of the ferrous α, α'-dipyridyl complex, an equivalent amount was sufficient for the extraction of the *o*-phenanthroline complex. The authors postulate the formation of compounds of the type $Fe(C_{10}H_8N_2)_3 . (SO_3R)_2$ and $Fe(C_{12}H_8N_2)_3 . (SO_3R)_2$ where R represents a long chain alkyl group.

Walter and Freiser[126] have examined the analytical aspects of the reactions of 2-(2′-pyridyl)-benzimidazole and 2-(2′-pyridyl) imidazoline with ferrous iron. Both reagents resemble *o*-phenanthroline in their reactions, giving intensely coloured compounds with Cu^{I}, Cu^{II}, Co^{II}, Fe^{II} and Fe^{III}. The extraction of the ferrous complex of 2-(2′-pyridyl) imidazoline takes place from perchlorate solution only, but the 2-(2′-pyridyl) benzimidazole complex can be extracted into isoamyl alcohol from aqueous solutions at any pH. Amounts of iron from 0·5–500μ g. were extracted.

The isonitrosoacetophenone complex of ferrous iron, which is green in colour, can readily be extracted from aqueous solutions at pH 8–9. Unfortunately, very many metals are extracted under almost identical conditions.[127, 128]

Cobalt

Only trace amounts of cobalt are extracted from aqueous 8N nitric acid by diethyl ether, and virtually none from 6N hydrochloric acid. Grahame and Seaborg[131] found the distribution coefficient to be 10^{-4} for trace amounts. Cobalt is not extracted from iodide, bromide or fluoride solution, but several papers deal with the solvent extraction of cobalt as the thiocyanate from aqueous solution. Several organic solvents may be used to extract the ammonium or potassium cobalt thiocyanate complex $K_2Co(CNS)_4$. Young and Hall[129] used a mixed solvent containing 3 parts of amyl alcohol and 1 part of ether to extract the cobalt, in developing a colorimetric method for determining the latter. The extraction was also studied by Bayliss and Pickering[130] who similarly used an amyl alcohol-ether solvent, the function of the ether being to depress the extractability of the ferric thiocyanate. Copper, zinc, and nickel extract more or less similarly to cobalt. Grahame and Seaborg[47] have studied the extraction of radio cobalt by the thiocyanate method, using amyl alcohol to remove ammonium cobaltothiocyanate from 10^{-5}–10^{-12}M solutions. In 10^{-5}M solution the distribution ratio was given as 10·05 and in 10^{-12}M solution as 10·9. A value of *ca.* 0·15 was assigned for the distribution ratio of ammonium cadmium thiocyanate under identical conditions.

Garwin and Hixson[131, 132] have studied the solvent separation of nickel and cobalt as the basis of a possible commercial method. Anyhdrous cobalt is soluble in organic solvents having polar functional groups containing oxygen. It may be that an actual hydroxyl group is responsible in the alcohols and acids, or a potential hydroxyl group (by enolisation) in the ketones and esters. Ethers exert no solvent action on anhydrous cobaltous chloride, and the solubility is much higher in alcohols than in ketones. Preliminary experiments by the authors revealed that extraction of cobaltous chloride by capryl alcohol only became appreciable when the cobalt solution was virtually saturated. The influence of various electrolytes on the extraction split these into three categories. Substances such as hydrochloric acid and calcium chloride produced a colour change in the aqueous cobalt solution from red to violet to dark blue, whilst electrolytes such as sodium and ammonium chloride produced a colour change to dark red. In the third category, salts such as sodium sulphate produced no alteration in colour, and indeed reversed the colour change produced by the first two categories. Lithium, magnesium, and aluminium chlorides produced colour changes, whilst zinc, tin^{II}, and cadmium chlorides produced the opposite effect. Cobalt is extracted from the dark blue solution and not at all from the red solution. Several hypotheses have been proposed to explain the colour change produced on addition of various electrolytes, such as the formation of complexes and the hydration theory. Garwin and Hixson favour the hydration theory, on the grounds that the solvents which extract the cobalt chloride in the presence of the

electrolytes which produce a blue colour are those which dissolve the anhydrous salt.

Separation factors varying from 40 to 90 and from 10 to 17 were obtained for the cobalt/nickel system using capryl alcohol with addition of hydrochloric acid and calcium chloride, respectively. The distribution coefficient of nickel chloride also increases with addition of electrolytes, but much more slowly.

According to Furman, Mason and Pekola,[17] the rose coloured compound formed by cupferron with cobalt^{II} is soluble in ethyl acetate and ether, with the formation of a deep red colour. Moeller found that the extraction of cobalt by oxine and chloroform began at *p*H 3·5, but only became complete above *p*H 6·8. Gentry and Sherrington[16], using a stronger reagent, obtained complete extraction over the range *p*H 5·7–9·5. Sandell[29] records that the optimum *p*H for extraction of the dithizonate by carbon tetrachloride is *p*H 7–9, and he remarks that the dithizonate in the extract is quite stable to dilute mineral acids. Chilton[133] has recently studied the extraction of the diethyl-dithiocarbamates of copper, cobalt, and nickel. Three extractions with carbon tetrachloride were found to remove completely microgram amounts of cobalt from solutions at *p*H 8·5–9·0. The presence of cyanide prevented extraction. The grey-brown complex of cobalt and *o*-nitrosophenol can readily be extracted by means of petroleum ether, with iron^{III} forming the main contaminant. The interference of iron can be overcome by adjusting the *p*H to 4, and adding sufficient tartrate or citrate to complex all the iron present. The *α*-nitroso-*β*-naphtholate of cobalt is also readily soluble in organic solvents such as chloroform.

Nickel

Nickel remains completely in the aqueous phase when a solution of the chloride in 6N hydrochloric acid is shaken with diethyl ether. Similarly, only minute amounts are transferred to the ether layer in extracting 8N nitric acid solution. The bromide, iodide and fluoride are almost completely unextracted under the conditions favouring extraction of other metals, and, indeed, the only purely inorganic compound of nickel which may be solvent extracted from aqueous solution is the thiocyanate. The conditions favouring the extraction of the thiocyanate are very similar to those for cobaltothiocyanate.[130] The compound formed between nickel, pyridine, and thiocyanate is also readily extracted by solvents such as chloroform.

The well-known nickel dimethylglyoxime precipitate is readily soluble in chloroform, and use of this chelating agent gives a very elegant method for concentrating small amounts of nickel from large volumes of aqueous solution. From an ammoniacal medium containing citrate, the solvent extraction gives a good separation from iron,^{III} cobalt, aluminium, etc. The separation from cobalt is noteworthy. Large amounts of cobalt do, however, interfere with the quantitative removal of nickel from the aqueous phase. Copper accompanies nickel into the chloroform. Sandell[29] comments that manganese interferes with the extraction of nickel, possibly by oxidising the nickel. Nickel^{III} dimethylglyoxime is not extractable. Back extraction of the nickel is simply achieved by equilibrating the extract with dilute hydrochloric acid. The dimethylglyoxime remains almost entirely in the organic phase.

Furman *et al*[17] comment on the solubility of nickel cupferrate in organic solvents, and it may be inferred that extraction is feasible. Nickel is amongst those metals which may be extracted as dithizonate. The extraction takes place from faintly alkaline solutions. The carbon tetrachloride extract is not readily decomposed by dilute mineral acid. The compound extracted is brown in colour, but a grey coloured product is extracted from strongly alkaline solutions.

Using 0·1M oxine in chloroform, Moeller[15] obtained quantitative removal of nickel from aqueous solution above *p*H 6·7. Partial extraction began at *p*H 2·5. Using a 1% reagent (0·7M) Gentry and Sherrington[16] found evidence for complete extraction in the range 4·5–9·5 in the presence of tartrate. Lacoste, Earing and Wiberley[101] report that quantitative extraction of the nickel^{II} complex with sodium diethyldithiocarbamate occurs over almost the entire *p*H range when chloroform is used as the solvent. Chilton[13] studied the carbon tetrachloride extraction over the *p*H range 8·5–9·0, obtaining complete recovery of nickel in five extractions, as opposed to three extractions for cobalt and copper.

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English Electric Dollar Order

A CONTRACT for the manufacture and delivery to site of two 200,000 kVA., three-phase, 60-cycle, 154/13·8 kV. transformers has been awarded to the English Electric Export and Trading Co., Ltd., by the Tennessee Valley Authority of Chattanooga, for the Reynolds Power sub-station. The value of the contract is approximately £200,000, and the transformers will be manufactured at the Company's Stafford Works. This is the third order to be placed with English Electric by the Tennessee Valley Authority, the previous two contracts cover equipment for the Rockwood and Johnsonville power stations.

New Automatic Grinding and Polishing Machine for Metallurgical Specimens

A METALLURGICAL microscope is only as good as the specimen preparation allows it to be. Possibly as a result of the realisation of the truth of this statement—possibly because specimen preparation and examination are both the business of the metallographer—a number of grinding and polishing machines for the preparation of metallurgical specimens have been designed and built by optical instrument makers. The most recent, the Beck No. 1600, has been developed by R. and J. Beck, Ltd., after numerous consultations with experienced metallurgists, both in industry and research, and incorporates features found desirable by many years of work on the preparation of metallurgical specimens for microscopy.

While compact enough to be used on a bench, the external dimensions being $22 \times 15 \times 8$ in., it is robust and capable of either hand or automatic grinding and polishing. All bearings are self-lubricating, the worm gear mechanisms are grease packed and sealed, and other potential sources of deterioration have been so designed that a very long period of use without any maintenance can confidently be expected. All vulnerable metal surfaces are either of stainless steel or are hard stove-enamelled, and considerable thought has been given to the general design to facilitate cleaning.

The rubberoid material Gaco has been used extensively in the equipment, because it is completely impervious to all oils and spirits and will not perish in the same way as rubber, although physically it has very similar properties.

The equipment is carried on a fabricated frame of robust construction, housed in a metal box with a heavy gauge top-plate, the major part of which constitutes a lid (shown raised in Fig. 2). On the fixed portion of the top-plate, a reservoir *A* with lid for lubricating fluid is fitted, from which the feed to the work is provided by a flexible Gaco pipe *F* connecting the reservoir to the centre pivot of the reciprocating specimen bracket *B*. From this unit, the lubricant, is fed on to the working area via a metal tube, which is adjustable for position. The rate of flow of lubricant is controlled by a tap on the top of the pivot spindle.

Fig. 1.—No. 1600 automatic grinding and polishing machine, with multiple fixed chuck.

The main spindle *I* carries the grinding or polishing disc, as required, and is driven by a worm and spindle mechanism connected in turn to the belt and pulley system *H*. The motor *E* has a rating of $\frac{1}{6}$th h.p. at a constant speed of 1,425 r.p.m., and the pulleys and drive reduce this to manually adjusted spindle speeds of 100, 200, 300 and 400 r.p.m. The motor also actuates the reciprocating specimen holder *B*, via a flexible drive and a worm gear *G*. The specimen is thus oscillated across the grinding or polishing disc at a constant rate of 30 cycles per minute.

The lid can be opened by releasing the lock *L* on the front of the machine, and is retained in the open position by a spring arm, thus giving access to the belt and pulleys and facilitating the change of discs. When the lid is raised, the electrical circuit is automatically broken and the lubricant flow ceases, safety measures whose value will be readily appreciated.

The discs are carried on the spindle *I*, fixing being provided by a plain fitting and thread. Two forms of disc are available, one for grinding, using abrasive papers or cloths of the appropriate size and grade, and the other for polishing, employing suitable cloth pads. The discs are $5\frac{1}{2}$ in. diameter, and the face of the grinding disc has grooves cut across it to assist in the clearance of lubricant and sludge from the working area. The abrasive paper is held firmly in contact with the disc by a peripheral clamp ring, which is secured by three pins on the disc, which fit into the helical slots in the ring. Rigid fixing is achieved by turning the ring about $\frac{1}{6}$th of a turn, at which point eight small Gaco pads press against the abrasive paper, sandwiching it firmly in position on the face of the polishing disc. The rim of the retaining ring is raised above the level of the grinding surface by approximately $\frac{1}{8}$ in., and holes are therefore cut through this raised rim to allow the lubricant and sludge to escape from the grinding surface.

The polishing disc has a plane top surface, and polishing cloths are held in contact with this by a Gaco ring, which serves to hold the stretched cloth. A groove is cut in the periphery of the metal disc to receive this retaining ring.

Extra weight can be added to the spindle *B* so that the pressure at the working surface can be controlled. Spare weights, supplied in 3, 6, 12 and 24 oz. units, are carried on the fixed pillar *C* fitted to the lid.

Three forms of specimen holder are available, the simplest being a single pin on the end of the spindle *B*, which drops into a hole cast into the specimen blank.

This enables the specimen to find its own level and results in quicker polishing, but in extreme conditions it can result in a slight rounding of the specimen edge. The rounding can be obviated by using the single fixed chuck, but since this prevents the specimen from being self-levelling, longer periods of grinding may be required. Also, for the fixed chuck, the specimen must be set in a 25 mm. diameter blank, whereas the single pin imposes no restriction on the specimen size.

Where large numbers of specimens are to be prepared, a three-specimen chuck is used. This is coupled to the oscillating head by the single pin already described, and accommodates three blanks 25 mm. diameter. This chuck enables identical polishes to be achieved on three similar specimens, which can then be independently etched.

An anti-splash ring *D* which surrounds the disc is fitted, and lubricant thrown off the rotating disc is caught and drains away into the catcher bowl *J*. This bowl is so shaped that fluid is prevented from reaching the main spindle, and is equipped with a drainage pipe through which the lubricant runs away into the lower reservoir *K*. An automatic tap is fitted to the end of the drainage pipe, so that, when the container is removed to recharge the upper reservoir, the flow of liquid is automatically stopped. The capacity of the upper container *A* is less than that of the lower *K*, so that overflow is prevented, but as a further safety measure an overflow outlet is cut in the endplate *M*, so that, should superfluous liquid escape, it appears at the front of the machine, rather than inside where damage may be caused. A filter paper along the top of the lower reservoir serves to remove the dirt from the lubricant, which can therefore be used again.

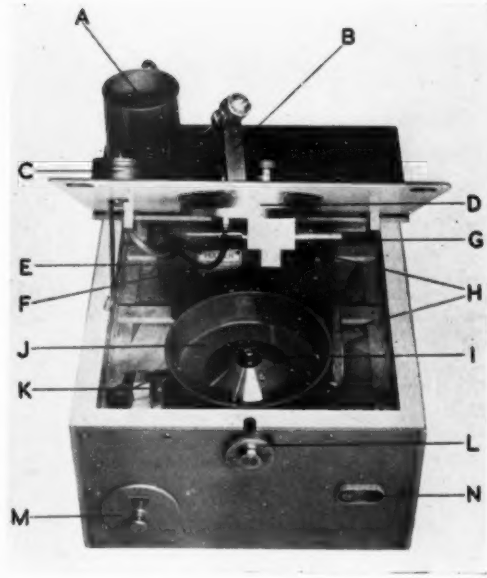

Fig. 2.—View of the machine with the lid raised.

A substantial plinth is available on which the machine stands, the working surface being the normal bench height of 30 in. The top surface is plain and can be easily cleaned, and a drawer for spare accessory equipment is incorporated. A simple " on-off " push switch for the electrical circuit is fitted to the front of the cabinet.

New Radiographic Installation

Plans for one of the most powerful and versatile X-ray installations in the metals industry have been disclosed by Chicago Bridge and Iron Company, builders of pressure vessels and heavy duty weldments for industry and the nuclear energy field. The proposed facility will house two complementary Van de Graaff super-voltage X-ray generators—1- and 2-million volts respectively—both manufactured by High Voltage Engineering Corporation, Cambridge, Mass. They will be used jointly in inspection of storage and pressure vessels and investigations of nuclear reactor chambers.

The most striking aspect of this installation is the unusual mounting planned for the 1-million-volt machine. This device will be fixed on the end of a 43-ft.-long counterweighted boom, mounted on rails, so designed as to fit easily inside vessels under test. When used in conjunction with the 2-million-volt machine, which will be operated from an overhead crane mount, the number of circumferential radiographic inspections will be materially increased, allowing the company to check nearly four times as many units as before. Both machines will be housed in a specially-built concrete structure, 42 ft. wide × 115 ft. long. Walls 4 ft. thick will be topped by a 2½-ft. thick roof. Doors at each end will simplify entry and exit.

According to High Voltage officials, this marks the most advanced application to date of supervoltage equipment for industrial radiography. They point out that the Van de Graaffs will enable a degree of diversity, speed, accuracy and economy in radiography not available with any comparable equipment. It is expected that this new installation will also enable Chicago Bridge to fabricate welded vessels with greater wall thicknesses than previously possible. The 2-million-volt unit provides a means for rapid and accurate inspection of steel thicknesses up to 10 in., with less than one minute exposure required for thicknesses under 5 in. This machine records, with better-than-average sensitivity, discontinuities thinner than ½% of total thickness, and a thickness range of slightly more than 2 in. of steel can be recorded on a single film with a single exposure. The sealed-off X-ray tube in the Van de Graaff features a titanium absorber system, which preserves the necessary internal vacuum without sacrificing low-cost construction techniques.

Henry Wiggin Purchase Hereford Plant

Henry Wiggin & Co. Ltd., announce that they have purchased the specialised high nickel alloy fabricating plant at Hereford, which they designed and erected for the Ministry of Supply and have been operating since 1954. The Company is a wholly-owned subsidiary of The Mond Nickel Co. Ltd., an affiliate of The International Nickel Co. of Canada, Ltd.

Chemical Research Laboratory Report for 1955

Atomic Energy Problems Influence Programme

THE present programme of research at the Chemical Research Laboratory covers such a wide range of subjects in relation to the staff available, says the Report* of the Chemistry Research Board for 1955, that effort is to be concentrated on a number of selected topics only. There is a tendency for outside organisations to call upon the Laboratory for testing services in connection with subjects and techniques that have ceased to be of research interest. Steps are being taken to ensure that this type of work is done by consultants and not by the Laboratory.

The report emphasizes the close link between the Laboratory, the U.K. Atomic Energy Authority, and the mining concerns which are extracting and concentrating metals valuable in the development of atomic energy. Nearly one quarter of the scientific staff of the Laboratory is employed on work for the Authority, the main projects being the study of minerals, ores and other materials, with a view to the extraction of valuable metals.

Extraction of Uranium and Other Precious Metals

The radiochemical group has intensified its efforts in the study of chemical extraction and concentration of metals. Samples of uranium ores from various parts of the world have been used in the development of methods of extraction of the metal on a large scale. The processes investigated include those which involve leaching followed by selective precipitation or recovery by ion-exchange or by solvent extraction.

The usual way of recovering metal from an ore by ion-exchange is to treat the ore with a solution which dissolves the metal, the metal being recovered from the filtrate after insoluble matter has been filtered off. A process in which the filtration step is left out is being investigated. The resin in suitable form is added to the pulp of ore and extracting solution. The ion-exchange can later be removed from the pulp mechanically and passed to an eluting circuit.

Major New Process for Gold Recovery

A new process for the recovery of gold from cyanide solutions has been developed which could revolutionize gold recovery technique. It has been found that gold and silver can be selectively absorbed from a normal gold mill pregnant liquor by certain weak base ion-exchange resins, and can then easily be eluted by a relatively inexpensive reagent from which it can be recovered by electrolysis. The gold-producing industry is showing great interest in the new process, although considerable research needs to be done before the method can be adopted commercially.

Separation of the Rare Earths

The rare earths are attracting increasing attention, partly because of atomic energy interests, and partly because they are no longer rare. The Laboratory is, therefore, engaged in a study of methods of separating the rare earths and of producing pure compounds and, ultimately, the pure metals. Ion-exchange methods are being employed, and an apparatus has been constructed capable of handling kilogram quantities of the mixed earths. This programme is of particular interest to the Atomic Energy Authority.

The Purification of Semi-conducting Elements

The vast research programme on semi-conductors has stimulated much work on the preparation of ultra-pure specimens of the elements showing such properties. Methods of preparing these materials are being studied by the Laboratory as a result of requests from a number of interested Government Departments.

Cellulose in Paper-strip Chromatography

One of the notable developments in chromatographic techniques made during the year has been the discovery that the usefulness of paper chromatography can be greatly increased by the use of chemically modified forms of cellulose. For instance, phosphorylated cellulose can absorb large quantities of certain metals from very highly acid solutions when normal ion-exchange resins would be ineffective.

The cellulose material can be used in the form of paper for strip chromatography or in the form of a floc for use in columns. A tendency for the material to deteriorate in storage has been overcome, resulting in a product sufficiently stable for practical purposes.

Ion-exchange Paper

Conventional methods for separations using ion-exchange resins are often unnecessarily elaborate for qualitative analysis. A simple method has been devised for preparing paper with ion-exchange properties in which a finely divided ion-exchange resin is incorporated in the cellulose pulp. The ion-exchange paper prepared from the pulp should be of value in qualitative analysis, and can be used for the separation of mixtures of cations (or anions), using techniques similar to those employed in conventional paper chromatography.

* "Chemistry Research 1955" is published by Her Majesty's Stationery Office for the Department of Scientific and Industrial Research. Price 4s. (72 cents U.S.A.), by post 4s. 3½d.

Anglo-U.S. Furnace Agreement

THE International Furnace Equipment Co., Ltd., of Aldridge, Staffs., have concluded an agreement to represent and manufacture the products of Holcroft & Co., of Detroit, Michigan. The agreement covers Great Britain, Australasia, and all British colonies and dominions except Canada, for heat treatment furnaces. Typical heat treating applications include carburising, carbonitriding, clean hardening, isothermal heat treating, and atmosphere generation.

1957 Physical Society Exhibition

THE Physical Society Exhibition in 1957 will be held in both the Old and New Halls of the Royal Horticultural Society, Westminster, London, S.W.1. The Exhibition will be open from 25th to 28th March inclusive.

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